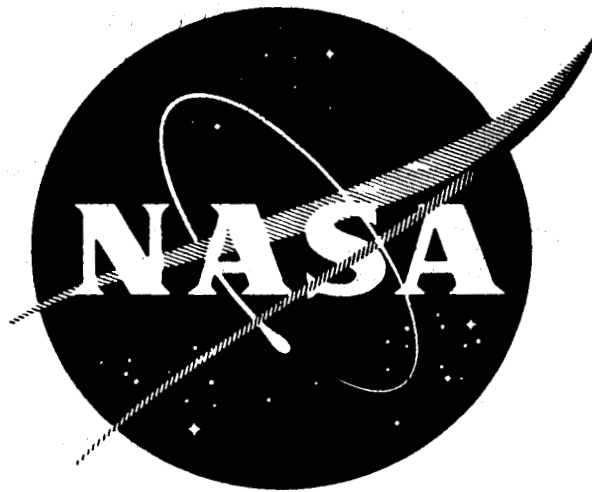


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EVALUATION OF HIGH STRENGTH COLUMBIUM ALLOYS FOR ALKALI METAL CONTAINMENT

FINAL REPORT

Covering the Period
July 25, 1962 to December 15, 1964

L. B. ENGEL, JR.
R. G. FRANK

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NAS 3-2140

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION

GENERAL  **ELECTRIC**
CINCINNATI, OHIO 45215

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FOREWORD

The work described herein was performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2140. Its purpose, as outlined in the contract, is to evaluate the corrosion resistance of high strength columbium alloys, to boiling and condensing potassium, as potential containment materials for space electric power conversion systems.

R.G. Frank, Manager, Physical Metallurgy, Materials and Processes, administered the program for the General Electric Company. Experimental investigations were performed by L.B. Engel, Jr., R.G. Carlson, and D.N. Miketta (deceased), with the assistance of W.H. Hendrixson, H.J. Bauer and D.R. Caldwell.

Mr. R.L. Davies, of the National Aeronautics and Space Administration was the Technical Manager for the study. Recognition is also extended to Mr. T.A. Moss for his assistance in monitoring the program.

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I. INTRODUCTION

Advanced turboelectric space power systems will require the containment of alkali metals at temperatures approaching 2200°F with possible hot spots at considerably higher temperatures. Only refractory alloys can be considered for the construction of such systems and, even then, critical components of the system will be strength limited. Consequently, there will be a reduction in system weight when the strongest alloy possible is used within the restrictions of system reliability. Recognizing the strength-weight requirement for service at temperatures up to 2200°F, in conjunction with the complex fabrication and joining involved in the construction of the system, it is apparent that columbium alloys have significant potential for accommodating these requirements in the near future.

Additionally, it is imperative to provide materials with suitable corrosion resistance to alkali metals and, although not fully documented, columbium alloys have also shown promising results in this respect. The only extensive experience obtained in the containment of alkali metals by columbium alloys preceding initiation of this program, however, had been with the relatively weak Cb-1Zr alloy, and it was generally recognized that a stronger alloy would offer a significant advantage for the advanced space electric power systems. This investigation was undertaken to document the behavior of AS-55 and D-43 columbium base alloys in an environment of refluxing potassium for periods up to 10,000 hr at temperatures of approximately 2000°F.

The AS-55 designation represents a columbium-base alloy with approximately 5% tungsten, 1% zirconium, and 0.06% carbon, which is arc melted with an addition of approximately 1% yttrium to the electrode (much of the yttrium removed as the volatile yttrium oxide (YO) during vacuum melting). The yttrium is added primarily to enhance both the fabricability, by the removal of oxygen during melting, and the weldability, by gettering of oxygen during welding. The D-43 designation represents a columbium-base alloy with approximately 10% tungsten, 1% zirconium, and 0.1% carbon. Inclusion of both alloys in the program permits the evaluation of two levels of carbon in the same alloy system and provides two alloys with varying strength/fabricability/weldability characteristics: i.e., the AS-55 alloy has a higher degree of fabricability and weldability; the D-43 alloy, a higher strength potential.¹ Also, both alloys contain reactive elements (zirconium, yttrium) which are strong oxide formers that react with the oxygen in solution in the columbium-tungsten base. Such alloys are expected to be compatible with alkali metals.

Both alloys are essentially in the columbium-tungsten-zirconium-carbon quaternary alloy system and achieve their superior strength by a combination of two basic strengthening mechanisms: solid solution of tungsten in a columbium matrix and dispersion of carbides formed by reactions between zirconium, columbium, and carbon. The use of the latter strengthening mechanism to increase the temperature capabilities of refractory alloys is considered generally to be very effective. However, the carbides that are formed during the processing of the material and that subsequently inhibit slip by impending dislocation glide must be chemically and thermally stable in the presence of potassium for a minimum 10,000 hr in the 2000°F to 2200°F temperature range. Determining the chemical and thermal stability of the complex carbides in AS-55 and D-43 alloys under these conditions, therefore, constituted one of the principal technical objectives of this program. The major objective, as discussed earlier, was the documentation of the corrosion resistance of these alloys to potassium at 2000°F over a period of 10,000 hours.

Eight corrosion capsules were tested and evaluated. One set of four capsules (two AS-55 alloy, one D-43 alloy, and one Cb-1Zr alloy) was exposed to refluxing potassium for 5000 hr; a second set of four capsules was similarly exposed for 10,000 hr. The results obtained from the 5000-hr tests were reported in Interim Report 2 (reference 2); the results of the 10,000-hr capsule tests are reported here. For clarity and continuity, many details of capsule preparation and testing procedures reported previously (reference 2) are either repeated completely or summarized in this report.

II. SUMMARY

Corrosion testing of four reflux capsules, two AS-55, one Cb-1Zr, and one D-43, containing purified potassium, was conducted at 2000°F for 10,000 hr in an ultra-high vacuum chamber capable of achieving a vacuum of 2×10^{-10} torr. A vacuum in the 10^{-8} to 10^{-9} torr range was maintained throughout testing. The condensing rate of the potassium contained within the one-in. diameter x 11-in. long capsules was approximately 37 ± 12 lb/hr/ft² at 2000°F.

Evaluation of the Cb-1Zr alloy capsule (No. 7) after the 10,000-hr exposure revealed a small amount of black deposit, accompanied by a gold discoloration of the metal around or near the deposits, on the inner surface of the capsule wall in the region between the primary condensing zone and the liquid zone. These black deposits were identified as essentially pure zirconium with a high oxygen content; some columbium was also present. The deposits probably resulted from zirconium and oxygen being leached from the primary condensing zone by the potassium condensate. Such leaching was substantiated by the small reduction in zirconium and oxygen content observed in the primary condensing zone with a corresponding grain growth to a depth of 6 mils on the inner surface of this zone. Similar zirconium deposits and grain growth effects were not observed in the AS-55 and D-43 alloy capsules.

Evaluation of the AS-55 alloy capsule (No. 9) revealed discoloration on the inner surface of the capsule wall in the region between the primary condensing zone and the liquid zone. The extent of the discoloration observed in the AS-55 alloy capsule (No. 9) was significantly less than that found in the Cb-1Zr alloy capsule (No. 7). A small amount of white, nonmetallic deposit, believed to be Y_2O_3 , was observed in localized areas on the inner surface of the AS-55 alloy capsule (No. 9).

An improved potassium transfer system was employed to fill the second AS-55 alloy capsule (No. 11) and the D-43 alloy capsule (No. 4) and no signs of staining, evidenced in the earlier capsules, were observed in either capsule after the 10,000-hr exposure. As in the case of AS-55 alloy capsule No. 9, however, a small amount of a white, nonmetallic deposit, believed to be Y_2O_3 , was observed in localized areas on the inner surface of AS-55 alloy capsule No. 11.

Chemical analyses of samples obtained from the capsule walls revealed no significant mass transfer of carbon with either the AS-55 or D-43 alloy. However, the results of the chemical analyses indicate that oxygen can be leached from the material located in the region where the freshly distilled potassium condenses.

Coalescence of the carbides in the grain boundaries apparently occurred in the AS-55 and D-43 alloys and, as expected, stress-rupture testing of specimens machined from the capsule walls after the 10,000-hr exposure revealed a significant decrease in strength. This is an indication of thermal instability that is not necessarily associated with the presence of potassium.

Overall, only a small amount of corrosion, consisting primarily of staining and black deposits of zirconium, the formation of yttrium compound believed to be Y_2O_3 , and leaching of oxygen from the capsule material by the potassium, was observed. The amount of staining and black deposits was either reduced or eliminated in those capsules which were filled using the improved potassium transfer system showing the absolute necessity of proper handling to reduce contamination in alkali metal systems. In general, the materials exhibited excellent corrosion resistance. There was no significant solution attack or penetration of the grain boundaries in either the weldments or the base metal of any of the alloys tested.

III. TECHNICAL PROGRAM

Materials Procurement

The General Electric Company produced the AS-55 alloy sheet used to manufacture capsules for this program. Three heats, NAS-555, NAS-5514 and NAS-5515, were utilized in the capsule preparation. Heats NAS-555 and NAS-5514 were consumably arc cast, forged, warm rolled and, subsequently, cold rolled to produce 0.080-in. thick sheet; heat NAS-5515 was consumably arc cast, extruded, warm rolled and, subsequently, cold rolled to 0.082-in. thick sheet.

The D-43 alloy sheet was procured from the E.I. duPont de Nemours Company. It was produced from an ingot which was consumably arc cast, extruded, warm rolled and finished by cold rolling to 0.080-in. thick sheet.

The Cb-1Zr alloy sheet was purchased from the Stellite Division, Union Carbide and Carbon Corporation. The ingot was electron beam melted, forged and cold rolled to 0.100-in. thick sheet.

Processing details of the heats are summarized in Appendix A. Chemical analyses of the finished sheets are given in Table I and the final heat-treating history is recorded in Table II.

Capsule Preparation

Eight capsules, two Cb-1Zr alloy, two D-43 alloy and four AS-55 alloy, were fabricated from the 0.080-in. thick sheet described above. The sheets were roll formed into cylinders 11 in. long and approximately one in. in diameter. A set of formed capsules, together with machined end caps and bend specimens, is shown in Figure 1. The heat-treatment histories of the capsules before forming, after forming, and after TIG welding are presented in Table II.

The TIG welding was performed in a chamber which was first evacuated to a pressure of less than 1×10^{-4} torr and then backfilled with helium which had been passed through a dry-ice trap. Before welding each capsule, a weld pass was made on a sheet of titanium to getter oxygen in the system and thereby minimize contamination of the weldments during the welding process. The filler material used during the welding was the same as the capsule material. Table III gives typical chemical analyses of such welds in Cb-1Zr alloy sheet; Figure 2 shows typical welded capsule tubes. Although weldments made in the manner described are sound and considered satisfactory for the intended application, significant improvements

Table I. Chemical Analyses of AS-55, Cb-1Zr, and D-43
Alloy Sheets Used in the Fabrication of Reflux Corrosion Capsules

Chemical Analyses, wt. %									
Alloy	Heat	Condition	W	Zr	Y	C ^a	O ^b	N ^b	H ^b
AS-55	NAS-555 ^c	Stress-relieved 2300°F/1 hr	5.35	0.81	0.73	0.059	0.0187	0.0143	---
AS-55	NAS-5514 ^c	Stress-relieved 2300°F/1 hr	5.25	0.78	0.02	0.058	0.0463	0.0226	0.0003
AS-55	NAS-5515 ^d	Stress-relieved 2200°F/1 hr	5.17	0.86	0.27	0.064	0.0210	0.0205	0.0003
Cb-1Zr	Stellite-519	Recrystallized 2200°F/1 hr	---	1.29	---	0.004	0.0144	0.0028	0.0005
D-43	DuPont-322	Stress-relieved 2200°F/1 hr	9.6	0.99	---	0.085	0.0041	0.0030	0.0001

^aAverage of two analyses; complete cross section of sheet used for sample.

^bAverage of two vacuum fusion analyses; complete cross section of sheet used for sample.

^cAnalyses of metallic elements performed on samples taken from as-rolled plate.

^dAnalyses of metallic elements performed on samples taken from ingot.

Table II. Thermal History of Reflux Corrosion Capsules Prior to Filling with Potassium

Capsule No.	Alloy	Ingot No.	Heat-Treatment, °F ^a		
			Before Forming	After Forming	After TIG Welding
6, 7	Cb-1Zr	Stellite-519	2200	2300	2200
8	AS-55	NAS-5514	2300	2800	2400
11, 12	AS-55	NAS-5515	2200	2800	2400
9	AS-55	NAS-555	2300	2800	2400
2, 4	D-43	D-43-322	2200	None	2400

^aAll heat-treatments performed for 1 hr in a vacuum of $<1 \times 10^{-4}$ torr.

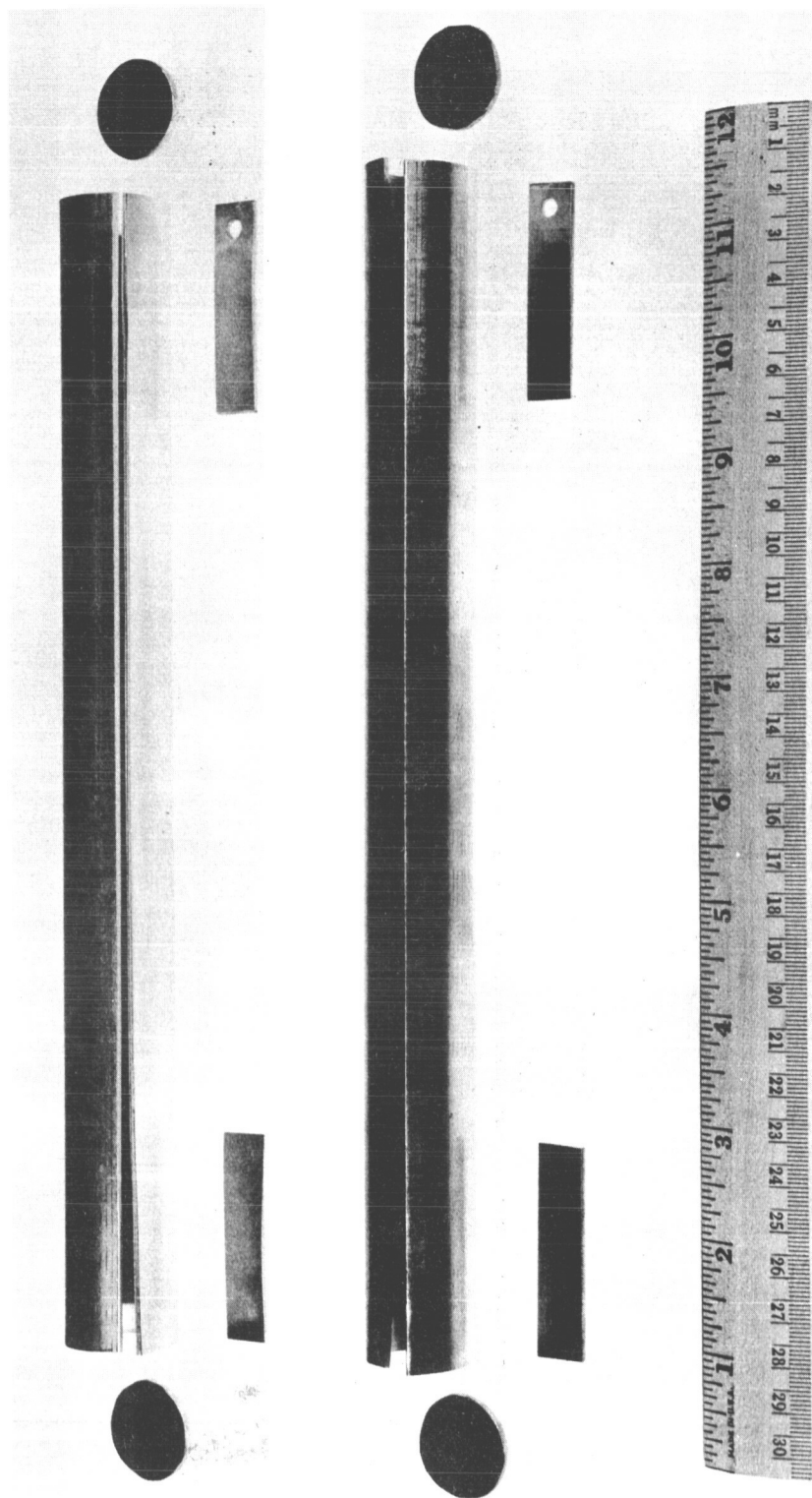
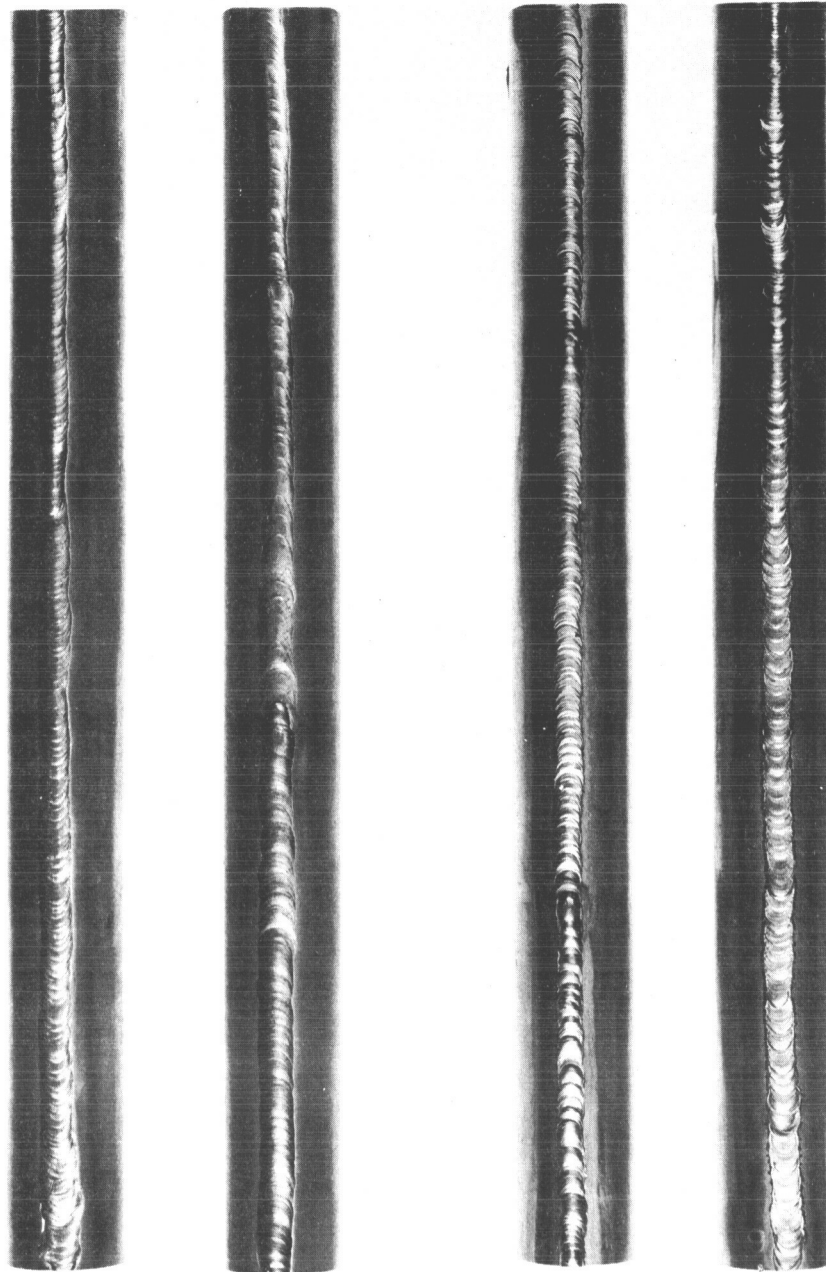


Figure 1. Typical Reflux Corrosion Capsules, End Caps, and Bend Specimens
Before Assembly and Welding: Top, AS-55 Alloy; Bottom, D-43
Alloy.

Table III. Chemical Analyses of Cb-1Zr Alloy Welds

Test	Sheet Thickness, in.	Sample Location	Chemical Analyses, ppm				Remarks
			C	O	N	H	
1	0.040	Weld	--	390	145	1	Chamber was vacuum purged once to 1 x 10 ⁻⁴ torr before filling with He passed through a dry-ice cold trap.
		Parent Metal	--	360	160	2	
2	0.060	Weld	--	126	27	4	
		Parent Metal	--	115	45	5	
3	0.025	Weld	80	406	78	-	
		Parent Metal	58	382	84	-	



Cb-1Zr
CAPSULE 6

Cb-1Zr
CAPSULE 7

AS-55
CAPSULE 8

AS-55
CAPSULE 9

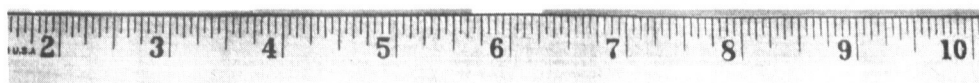


Figure 2. Formed and TIG Welded AS-55 and Cb-1Zr Alloy Reflux Corrosion Capsules.

have been made in TIG welding equipment and procedures for refractory alloys in the past several years, as evidenced by work conducted under recent NASA sponsored research and development contracts.³⁻⁶

Two fill tubes, 0.5-in. OD x 0.040-in. thick wall, were fabricated from each of the AS-55 and Cb-1Zr alloy capsule materials and TIG welded to the top end caps, also fabricated from the capsule material. Subsequently, the top end cap and fill tube subassemblies and the bottom end caps were TIG welded to the formed and welded capsule cylinders. In the case of the AS-55 alloy capsule No. 8 and capsule No. 9, short lengths of 0.500-in. OD x 0.040-in. thick wall, centerless ground Cb-1Zr alloy tubing were TIG welded to the top of the AS-55 alloy fill tube to facilitate crimping of the fill tube after filling with potassium. The length of Cb-1Zr alloy tubing was removed after the capsule was sealed. The capsule components were pickled in an acid solution of 20%HF+20%HNO₃+60%H₂O, rinsed in water, and given a final cleaning with ethyl alcohol before specimen placement and assembly welding. Figure 3 is a schematic of the AS-55 alloy capsule design. Except that it is monometallic, the Cb-1Zr alloy capsule design (capsule Nos. 6 and 7) is the same as that depicted for the AS-55 alloy capsule.

As shown in Figure 3, all four capsules (Nos. 6, 7, 8, and 9) contained 0.080-in. thick x 0.5-in. wide x 2-in. long bend specimens, made from the same material as their respective capsules, in the liquid and vapor regions. The interiors of the capsules, with bend specimens in position, were examined with a borescope and no detrimental defects could be observed in the welds. The capsules were leak checked with a helium mass spectrometer, as prescribed in MIL-Std-271B, and found leak tight. A radiographic examination of the four capsules demonstrated their welds to be sound. Figure 4 is a photograph of Cb-1Zr alloy capsule No. 6 and capsule No. 7 and AS-55 alloy capsule No. 8 and capsule No. 9 before filling with potassium.

Capsules 6, 7, 8, and 9 were charged with slagged, filtered, distilled and hot trapped potassium, as received from Mine Safety Appliance Research Corporation, using the facilities shown in Figure 5 and these outlined procedures:

1. Evacuate the capsule loading system to below 1×10^{-3} torr.
2. Heat the system to above 150°F to permit outgassing.
3. Close vacuum valve (A) to seal the capsule under a vacuum; close vacuum (B) to isolate the system from the vacuum pumps.
4. Flush the system, other than the capsule, with argon by opening valves (C and D).

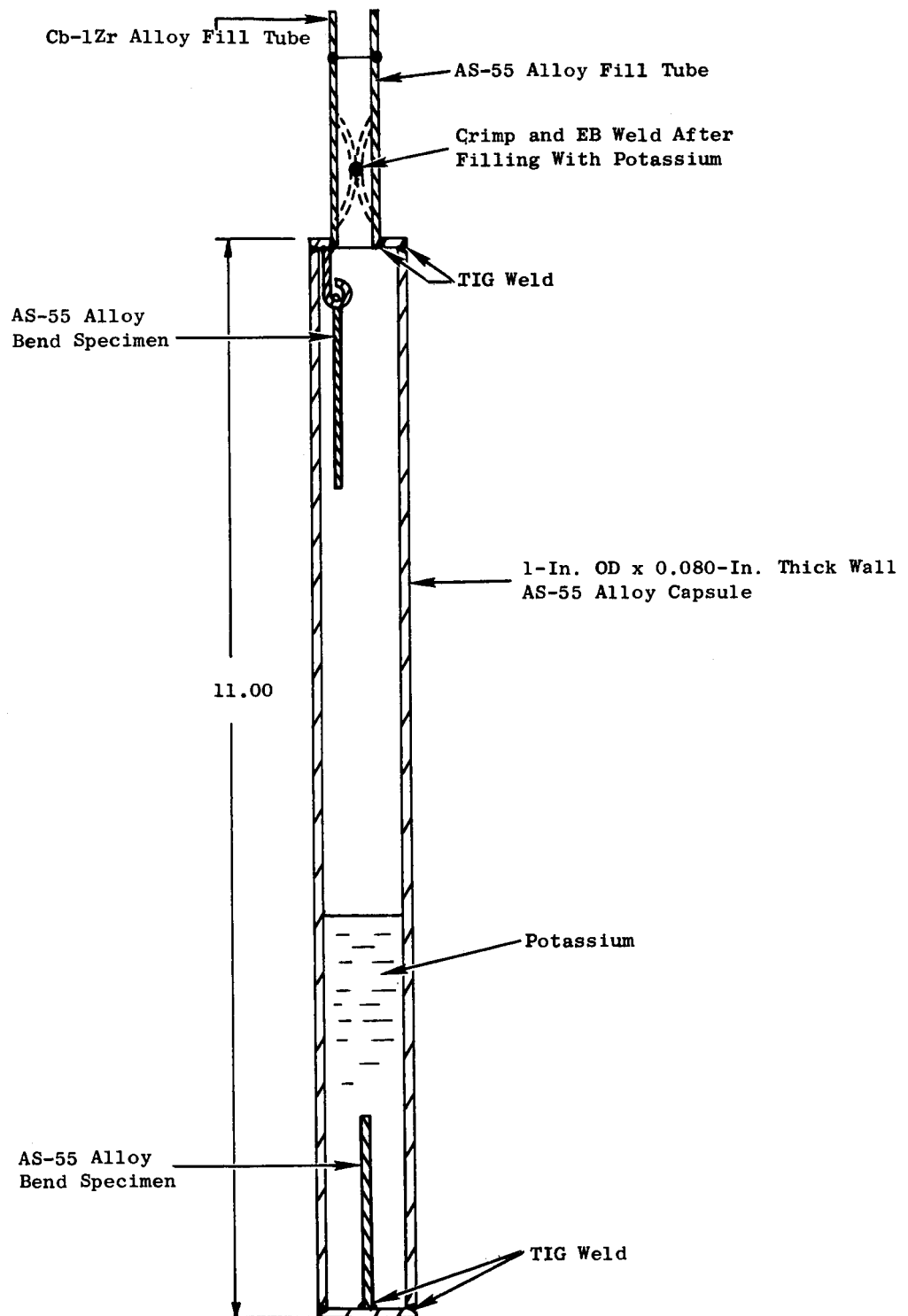


Figure 3. AS-55 Alloy Reflux Corrosion Capsule Showing Fill Tube and Location of Bend Specimens.



Figure 4. AS-55 Alloy Capsules Nos. 8 and 9 (left) and Cb-1Zr Alloy Capsules Nos. 6 and 7 (right) Prior to Filling with Potassium.

5. Re-evacuate the system to below 1×10^{-3} torr by closing valves (C and D) and opening valve (B).
6. Refill the system with argon.
7. Close one argon valve (D); leave other argon curtain flow valve (E) open.
8. Force potassium through the system by opening valve (F) and potassium storage container valve (G) until potassium flows from the argon curtain opening.
9. Allow the potassium to stay in the system 3 to 5 minutes.
10. Force new potassium into the system (see 8 above).
11. Close valve (C) to seal potassium in a sampling tube; close valve (F) to confine a measured amount of potassium.
12. Open capsule valve (A); simultaneously open argon valve (D) to force the measured length of potassium into the capsule (H).
13. Seal the capsule by closing valve (A), pinching down the tubing and welding in a vacuum by electron beam.

Table IV lists the chemical analysis of the slagged, filtered, distilled, and hot trapped potassium used to fill capsules No. 6, No. 7, No. 8, and No. 9 as obtained from the vendor. The potassium was analyzed for metallic elements by the Nuclear Materials and Equipment Corporation using spectrographic techniques and for oxygen by MSA Research Corporation using the mercury amalgamation method. The potassium was sampled at the same time and under the same conditions that the four reflux capsules, No. 6, No. 7, No. 8, and No. 9, were filled and was analyzed for oxygen by the zirconium-gettering technique. In this technique, a 0.005-in. thick corrugated zirconium strip, of known chemistry, with a total 20 in.² surface area, was inserted in a one-in. OD x 0.080-in. thick wall x 6-in. long Cb-1Zr alloy capsule. The capsule was filled with 7.14 grams of potassium, using the facility illustrated in Figure 5, sealed under vacuum by electron beam welding techniques, and heated for 100 hr at 1400°F in a vacuum of 10^{-5} torr. After exposure, the zirconium strip was removed from the capsule and analyzed for oxygen, nitrogen, hydrogen and carbon. From the interstitial analyses, and assuming that the interstitial elements completely reacted with the zirconium and no gaseous pickup occurred during the transfer of the potassium, the oxygen and carbon contents of the potassium were calculated to be 760 ppm (an average of four analyses) and 133 ppm, respectively. The data are presented in Table IV (ff.) and Table I (Appendix B). No significant change in the nitrogen or hydrogen could be detected in the zirconium getter material.

Oxygen analyses of the potassium used to fill capsules No. 6, No. 7, No. 8, and No. 9 also were obtained by the mercury amalgamation method at General Electric. In this case, however, the samples were drawn at

Table IV. Chemical Analyses of Potassium

Sample Identification	Date Analyzed	Chemical Analyses, ppm																							Remarks	
		0	Fe	B	Co	Mn	Al	Mg	Sn	Cu	Pb	Cr	Si	Ti	Mo	V	Be	Ag	Nb	Ba	Ca	Sr	Zr	Ni		
Shipping Container No. A-4																										
Vendor analysis	2-1-62	<100 ^b	50	<1	<10	3	1	20	2	<1	1	1	100	<5	<2	<5	<5	<1	75	2	15	1			Si and Ca levels are high due to contamination from glass sample tube.	
Capsule Filling ^c - Capsule Nos. 6, 7, 8 and 9																										
Sample cast in capsule direct from container	12-5-62	760 ^d																							Sample taken directly in evacuated Cb-12r capsule at time of filling of capsule Nos. 6, 7, 8 and 9.	
Sample obtained from transfer line	12-5-62	7 ^b																							Sample taken in evacuated and valved stainless steel pipe after filling capsule Nos. 6, 7, 8 and 9.	
Sample obtained from transfer line	12-5-62	16 ^b																								
Shipping Container No. 137																										
Vendor analysis	1-2-63	17 ^b	<5	<10	<5	<1	5	4	<6	<1	<5	<5	40	<5	<3	<1	<1	<1	45					<10	<5	Sample obtained in stainless steel pipe open on one end to flowing argon and capped in air.
GE analysis	1-23-63	141 ^b	-----																							
GE analysis	1-28-63	190 ^b																								
GE analysis	1-28-63	200 ^b																								
GE analysis	1-28-63	219 ^b																								
GE analysis	2-6-63	426 ^d																								
GE analysis	2-6-63	406 ^d																								
GE analysis	10-15-64	53.7 ^b	-----																							
GE analysis	10-15-64	52.4 ^b																								
GE analysis	10-16-64	44.3 ^b																								
GE analysis	10-16-64	50.8 ^b																								
General Atomics	10-5-64	83.5 ^e																								
General Atomics	10-5-64	73.1 ^e																								
NUMEC	3-10-63	-----	60	<10	<5	3	3	15	<5	3	<5	20	65	<5	<3	<1	<1	<1	100					<10	15	Sample obtained in evacuated stainless steel pipe and capped in air.
After Hot Trapping ^f 200 hr at 1300°F	3-11-63	20.8 ^b	25	<10	<5	<1	5	9	<5	3	<5	5	50	<5	<3	<1	<1	<1	100					<10	5	Sample obtained in stainless steel pipe open on one end to flowing argon and capped in air.
Capsule Filling ^g - Capsule Nos. 11, 12, 2 and 4																										
Sample cast inside tank	4-15-63	33 ^b																								
Sample cast inside tank	4-15-63	36 ^b																								
Sample cast inside tank	4-15-63	33 ^b																								
Sample cast inside tank	4-15-63	22 ^d																								
Sample obtained from transfer line	4-15-63	39 ^b																								
Sample obtained from transfer line	4-15-63	53 ^b																								

^aAll metallics determined from KCl by spectrographic techniques by the Nuclear Materials and Equipment Corporation.^bOxygen as K₂O determined by the mercury amalgamation method.^cPotassium from Shipping Container No. A-4.^dOxygen determined by zirconium-gettering technique (related data reported in Appendix B).^eOxygen determined by neutron activation at General Atomics.^fPotassium from Shipping Container No. 137.^gPotassium from Shipping Container No. 137 after hot trapping for 200 hr at 1300°F.

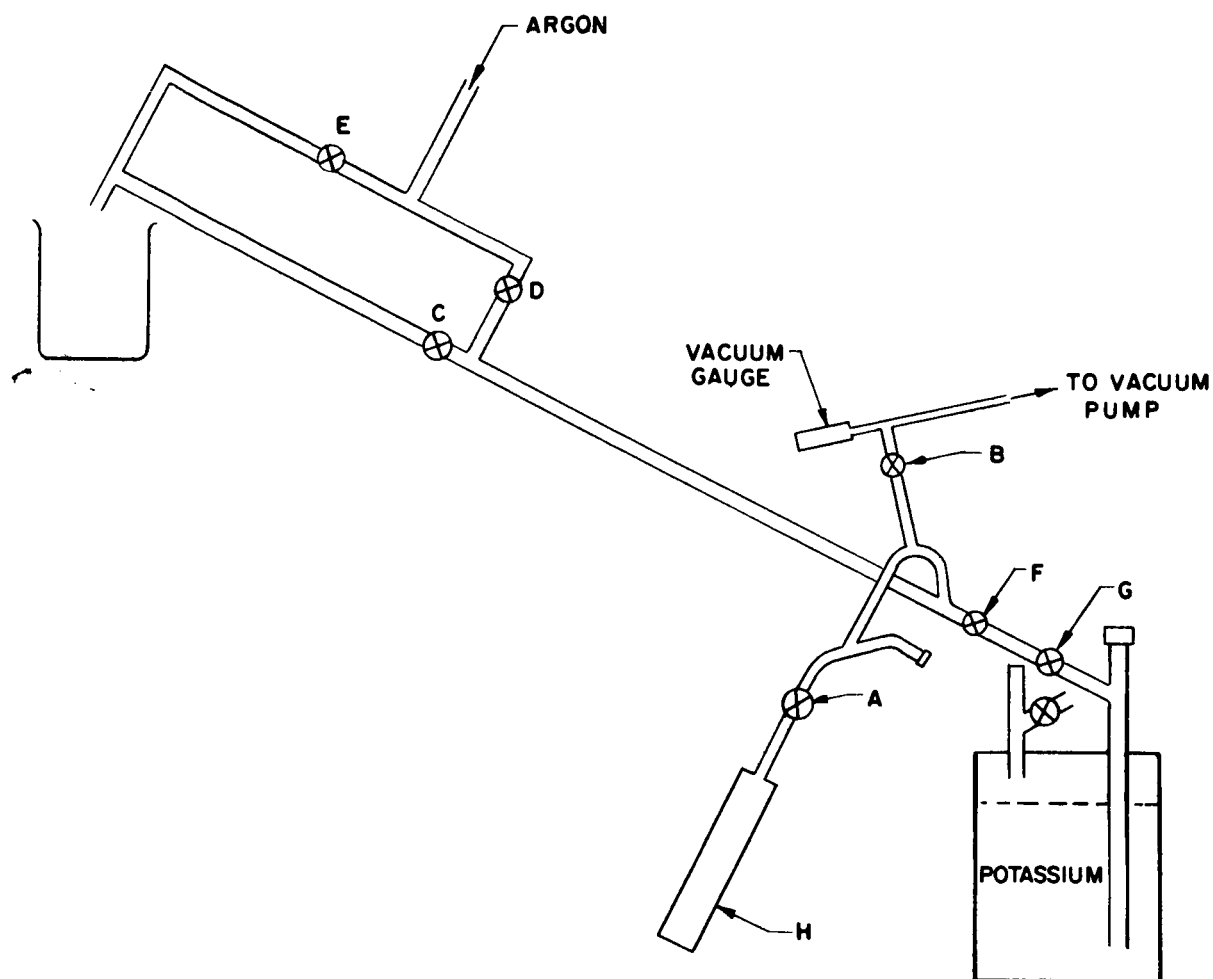


Figure 5. Apparatus for Filling Capsules Nos. 6, 7, 8, and 9 with Potassium.

a later date. The samples were taken by removing sections of tubing between valves G and F of the filling apparatus shown in Figure 5. Analysis of these samples produced values of 7 and 16 ppm oxygen as K_2O , Table IV. These data indicated that contamination was occurring during the filling of the capsules. To preclude this problem, a new potassium transfer facility was successfully developed and subsequently was used in filling the remaining two AS-55 alloy capsules (No. 11 and No. 12) and the two D-43 alloy capsules (No. 2 and No. 4).

The potassium transfer facility developed is similar to the transfer facility at NASA Lewis Research Center and consists of a 30 kv, electron beam welding chamber incorporating facilities for transferring potassium directly from the hot trap to the capsules in a vacuum of 5×10^{-5} torr with a leak rate of 1.4 micron-cubic feet per hour. The pumping system comprises a 10-in. oil diffusion pump and a 80 cfm Stokes Microvac mechanical forepump. The vacuum is measured with a National Research Corporation Type 501 ionization gauge and Model 710B control circuit. Figure 6 is an external view of the facility with the hot trap in place; Figure 7 shows the general capsule arrangement inside the chamber. The potassium is transferred to the capsules by pressurizing the hot trap, filling the stainless steel ladle with the potassium to the required level (30 grams), and then pouring the potassium through the stainless steel funnel into the capsule using a vacuum rotary feedthrough. After the capsule lid is manually placed on the capsule with the manipulator, the capsule is automatically positioned under the electron beam gun on the motor driven carriage and the lid is seal-welded to the top of the capsule as the capsule is rotated on its own axis. A series of six capsules can be filled without breaking the vacuum.

The remaining two AS-55 alloy capsules (No. 11 and No. 12) and the two D-43 alloy capsules (No. 2 and No. 4) were TIG welded, leak checked, and cleaned in a similar manner to that employed for the first two AS-55 alloy capsules (No. 8 and No. 9) and the two Cb-1Zr alloy capsules (No. 6 and No. 7) except that fill tubes were not required on these capsules. Bend specimens, 0.060-in. thick x 0.5-in. wide x 2-in. long, were also placed in these capsules. Subsequently, the four capsules were charged with slagged, filtered, and hot trapped potassium from Shipping Container No. 137 using the newly developed vacuum filling system.

Analyses of the as-received potassium from Container No. 137 used to fill capsules No. 11, No. 12, No. 2, and No. 4 were supplied by the MSA Research Corporation and are shown in Table IV. Before filling the corrosion test capsules, six additional analyses of the as-received potassium for oxygen (two by the zirconium-gettering technique previously described and four by the mercury amalgamation method) were performed at General Electric. These data are listed in Table IV. The method of sampling used for these analyses consisted of filling

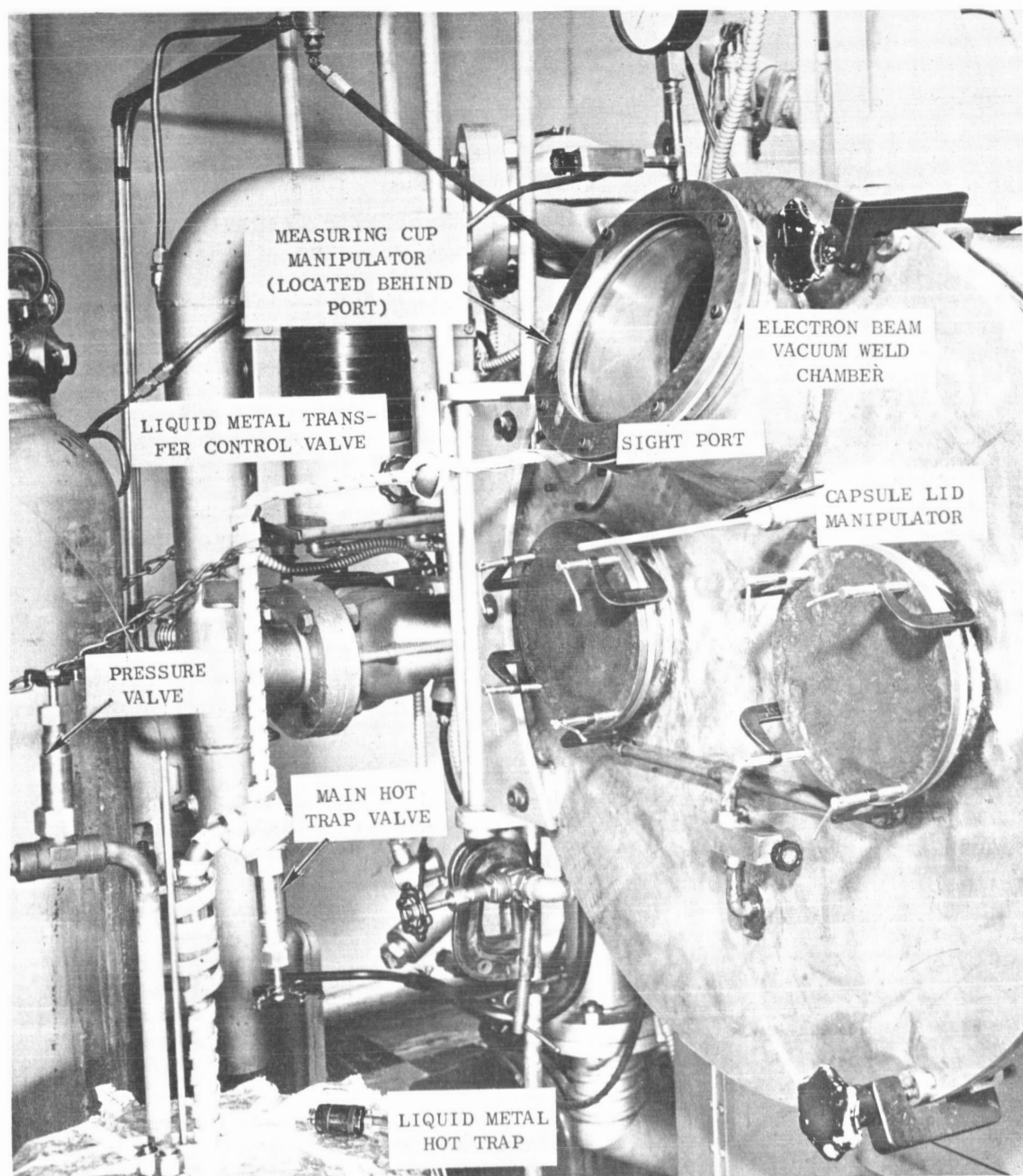


Figure 6. External View of the System for Alkali Metal Transfer (Hot Trap is Shown Attached to the Vacuum Tank Door).

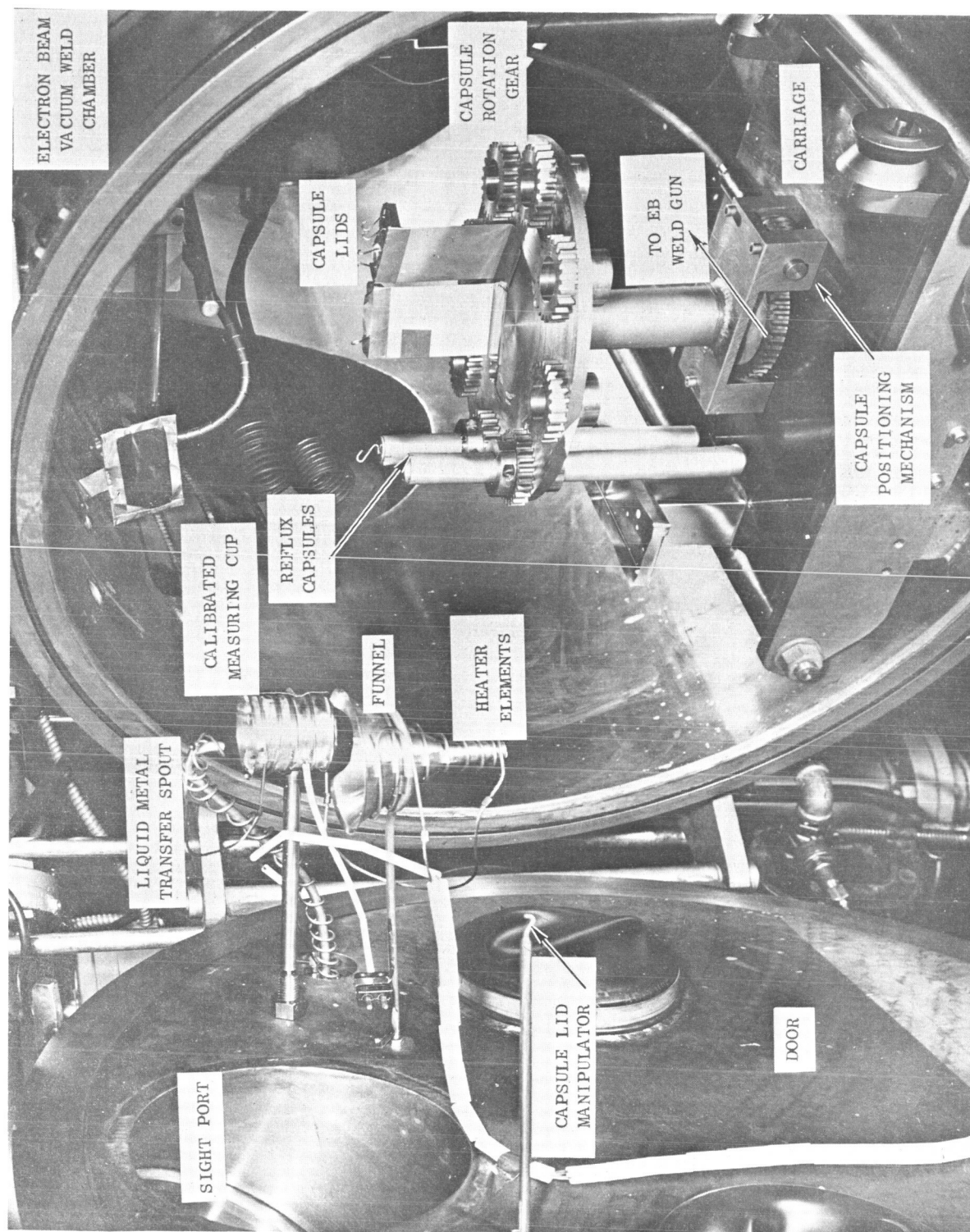


Figure 7. Internal View of System for Alkali Metal Transfer Showing Necessary Components for Filling Reflux Capsules (System Used for Filling Capsules Nos. 2, 4, 11, and 12 with Potassium).

three separate lengths of 0.5-in. diameter stainless steel pipe. One end was attached to the shipping container and the open end was exposed to flowing argon. After allowing approximately 250 grams of potassium to flow through the pipe, the contained potassium was permitted to solidify in two of the pipes and the ends were capped in air. Valves had been installed on each end of the third length of pipe and here the sample was obtained without exposing the ends to air. This pipe and one of the pipes capped in air were transferred to the electron beam welding facility and, after evacuation and back-filling with argon, the potassium was melted and cast into the Cb-1Zr alloy gettering capsules which were subsequently sealed under vacuum by electron beam welding. The potassium in the remaining length of pipe capped in air was analyzed by the mercury amalgamation method. The oxygen content determined by the zirconium-gettering technique, again consistently higher than that determined by the amalgamation method, is attributed to contamination from oxygen in the argon used in the welding chamber during transfer of the potassium to the gettering capsules. Note that there is little difference in the oxygen results obtained by the zirconium-gettering method as a function of the sampling method. The oxygen values from both methods, however, are considerably higher than the 17 ppm reported by MSA.

To assure that high purity potassium be used in the remaining capsule corrosion tests, approximately 16 lb of the as-received potassium from Container No. 137 were re-hot trapped at General Electric in a titanium-lined, zirconium-gettered container for 200 hr at 1300°F before being used to fill the capsules. The oxygen content of the potassium sampled from the hot trap after the hot trapping operation was approximately 21 ppm as analyzed by the mercury amalgamation method.

Additional chemical analyses of metallic impurities were also obtained on the potassium from Shipping Container No. 137 at an independent laboratory both before and after the 200-hr, 1300°F hot trapping operation; these values are recorded in Table IV.

In an independent study at this laboratory to evaluate the accuracy of the mercury amalgamation method for oxygen in potassium, three samples were obtained from Shipping Container No. 137 in lengths of stainless steel pipe which had been evacuated and outgassed, before filling with potassium, and subsequently capped in air. One sample was analyzed at General Electric by the mercury amalgamation method; two were sent to General Atomics to be analyzed by the neutron activation method where the potassium was melted and transferred to the copper capsules under purified helium.⁷ The results, shown in Table IV, are in relatively good agreement. It should be noted that the handling procedures used in obtaining the earlier samples from Shipping Container No. 137 are suspect and, for this reason, the latter results are believed to be more representative of the actual oxygen content of the as-received potassium.

To determine whether the re-hot trapped potassium was contaminated during the transfer and filling of the reflux corrosion capsules using the vacuum capsule filling facility, two samples were taken for the analyses of oxygen while the capsules were filled. One sample tube was filled with potassium inside the chamber in the same manner in which the corrosion capsules were filled; the other potassium sample was taken from the transfer line outside the chamber immediately above the hot trap container. Results obtained on these samples by the mercury amalgamation method are listed in Table IV.

A one-in. diameter x 10-in. long ancillary Cb-1Zr alloy capsule containing zirconium getter material was also charged with potassium during the actual filling of the corrosion capsules and sealed in an identical manner to that used for the test capsules and the sample tube used for the mercury amalgamation analyses. After a 100-hr exposure at 1400°F, the zirconium getter in the ancillary capsules was analyzed for oxygen, nitrogen, and hydrogen by the vacuum fusion techniques. From these data, recorded in Table IV of this section of the report and Table III of Appendix B, the calculated oxygen level in the potassium was found to be 22 ppm, which is in good agreement with the results obtained by the mercury amalgamation method, i.e., 33 ppm to 50 ppm. Therefore, the oxygen content of the potassium in capsules No. 11, No. 12, No. 2, and No. 4 was presumably less than 50 ppm.

After being filled with approximately 30 grams of potassium, all eight capsules were enclosed in tantalum foil and pre-tested in a vacuum of approximately 4×10^{-5} torr at 2000°F for 15 minutes to insure capsule integrity. Subsequently, the eight capsules were grit blasted with -280 mesh alundum powder, using an air pressure of 50 psig, to obtain a high surface emittance.

Testing Procedures

Three tantalum-clad, Pt vs Pt+13%Rh, Al_2O_3 insulated thermocouples* were attached to each capsule: one at the top; one at the radiating zone; one on the bottom immediately below the heater zone. Thermocouple placement, heater location, and radiation shielding are shown in Figures 8 and 9.

Four instrumented reflux capsules (No. 6, No. 7, No. 8, and No. 9) and their respective heaters and shielding were secured in a Varian ultra-high vacuum chamber C-III. This type vacuum equipment consist of an 18-in. diameter x 30-in. high bakeable chamber connected to a

* Purchased from Thermo Electric Company, Inc. Typical purity of the Al_2O_3 is 99.65% with the following maximum impurity levels: SiO_2 , 0.08%; Fe_2O_3 , 0.10%; Na_2O , 0.06%; MgO , 0.08%; CaO , 0.08%; ZrO_2 , 0.08%; C, 0.01%; B, 0.0001%.

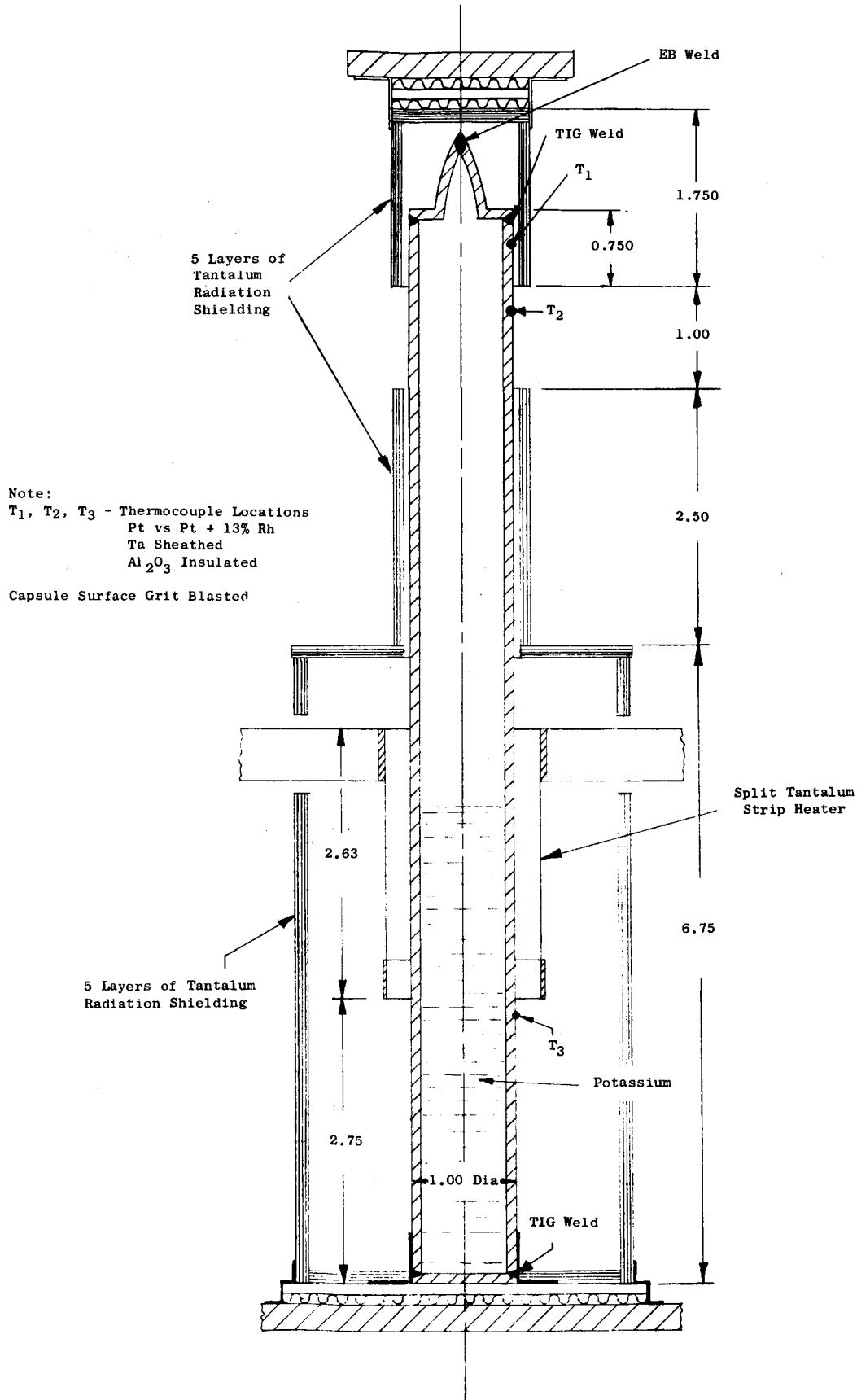


Figure 8. Drawing of Reflux Capsule Illustrating Location of Radiation Shielding, Radiation Zone, Tantalum Heating Elements, and Height of Potassium at Temperature in Capsules Nos. 6, 7, 8, and 9.

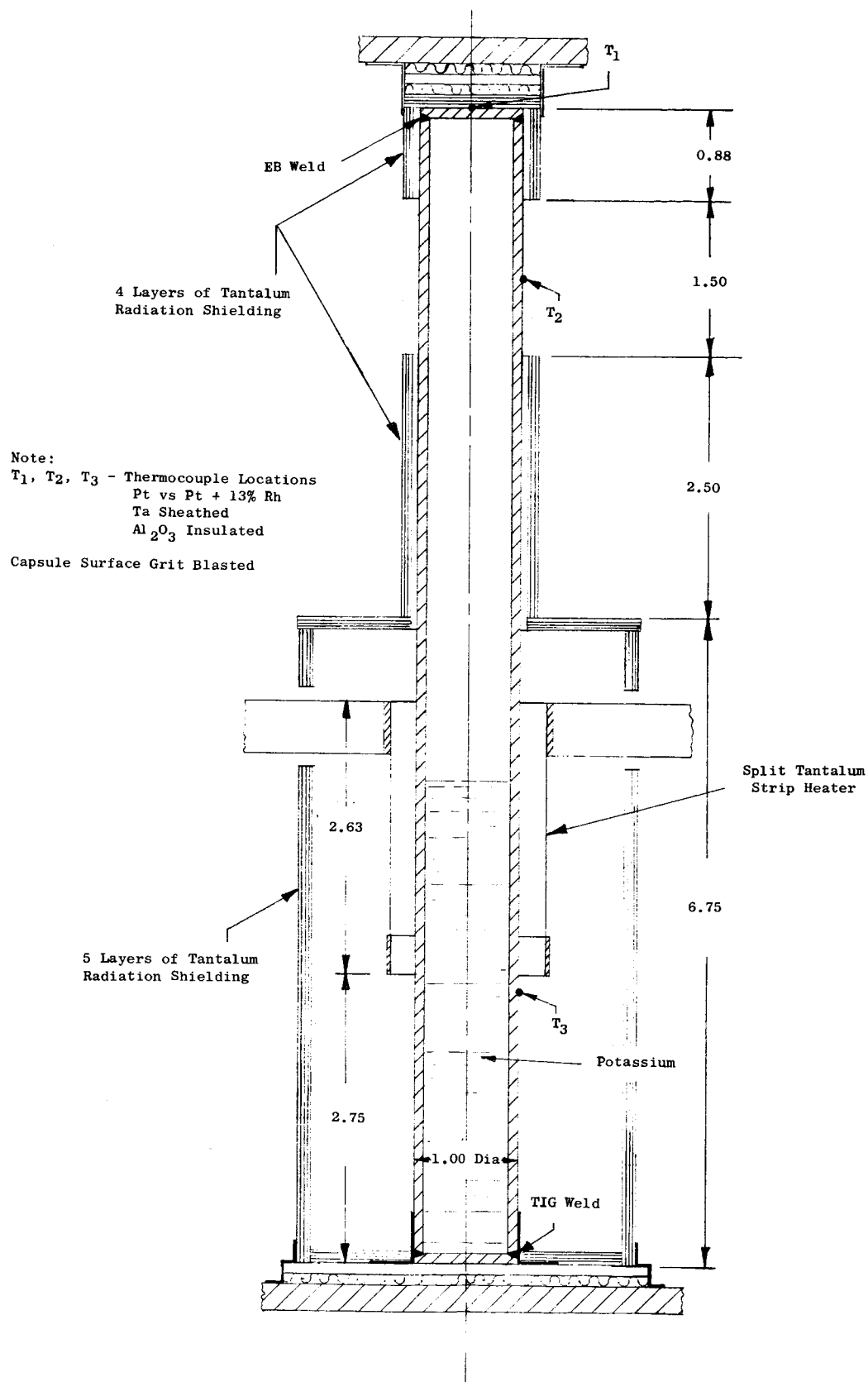


Figure 9. Drawing of Reflux Capsule Illustrating Location of Radiation Shielding, Radiation Zone, Tantalum Heating Element, and Height of Potassium at Temperature in Capsules Nos. 2, 4, 11, and 12.

400 ℓ /sec getter-ion pump and three cryogenic molecular sieve roughing pumps (Figure 10). Initial evacuation of the chamber to approximately 2 torr is accomplished with a mechanical pump and, subsequently, to the 10^{-3} torr range with the cryogenic adsorption pumps. Final evacuation to the ultimate pressure in the 10^{-10} torr range is accomplished with the getter-ion pump after a suitable bakeout period. Pressure is measured by a Bayard-Alpert type ionization gauge attached to the side of the chamber.

After installing capsules No. 6 and No. 7 (Cb-1Zr alloy) and No. 8 and No. 9 (AS-55 alloy) in the vacuum chamber, the chamber was evacuated to a pressure of 7.0×10^{-7} torr and baked out at 400°F for 8 hr after which the pressure reached 7.0×10^{-7} torr. Subsequently, the capsules were heated to 2000°F with the pressure being held below 3.0×10^{-6} torr during the entire heat-up cycle. Although some unstable boiling was evident during heat-up, the instability appeared to subside when the test temperature was reached. The four capsules were held at the test temperature of 2000°F for 5000 hr.

A pressure rise was encountered at the end of approximately 4500 hr when the coolant water to the vacuum bell jar was accidentally turned off. The pressure reached a maximum of 7.0×10^{-7} torr for a short time and dropped quickly when the coolant water was restored. At the end of 5000 hr exposure, the chamber was opened and capsules No. 6 (Cb-1Zr alloy) and No. 8 (AS-55 alloy) were removed from the chamber and evaluated. The results were reported in Interim Report 2.² Capsules No. 7 (Cb-1Zr alloy) and No. 9 (AS-55 alloy) resumed testing at 2000°F to complete the 10,000 hr. Two pressure excursions occurred during the last 5000 hr. After 5900 hr of testing, a pressure rise was encountered when a transformer in the getter-ion pump power supply failed. Although a maximum pressure of approximately 1×10^{-7} torr was reached for a brief period, the pressure dropped quickly when a portable getter-ion pump power supply was incorporated in the system. The faulty power supply was repaired and put back in service without further interruptions in testing. The second pressure rise was encountered after approximately 8950 hr of testing when the main power switch for the getter-ion pump was accidentally turned off. The pressure reached a maximum of 8×10^{-7} for a short period but dropped quickly when the switch was turned back on. The pressure profile for capsules No. 7 (Cb-1Zr alloy) and No. 9 (AS-55 alloy) over the 10,000-hr test period is shown in Figure 11.

Reflux capsules No 11 and No. 12 (AS-55 alloy) and No. 2 and No. 4 (D-43 alloy) were installed in the Varian ultra-high vacuum chamber C-II, which is similar to chamber C-III, and instrumented in the same manner as capsules No. 6, No. 7, No. 8, and No. 9. The chamber was evacuated to a pressure of 5.0×10^{-7} torr, as measured by a Bayard-Alpert type ionization gauge attached to the side of the chamber. During heat-up to the test temperature of 2000°F , the pressure reached



Figure 10. Varian Ultra-High Vacuum Systems Used for Capsule Corrosion Tests; Units Equipped with 400 and 1000 l/Sec Getter-Ion Pumps.

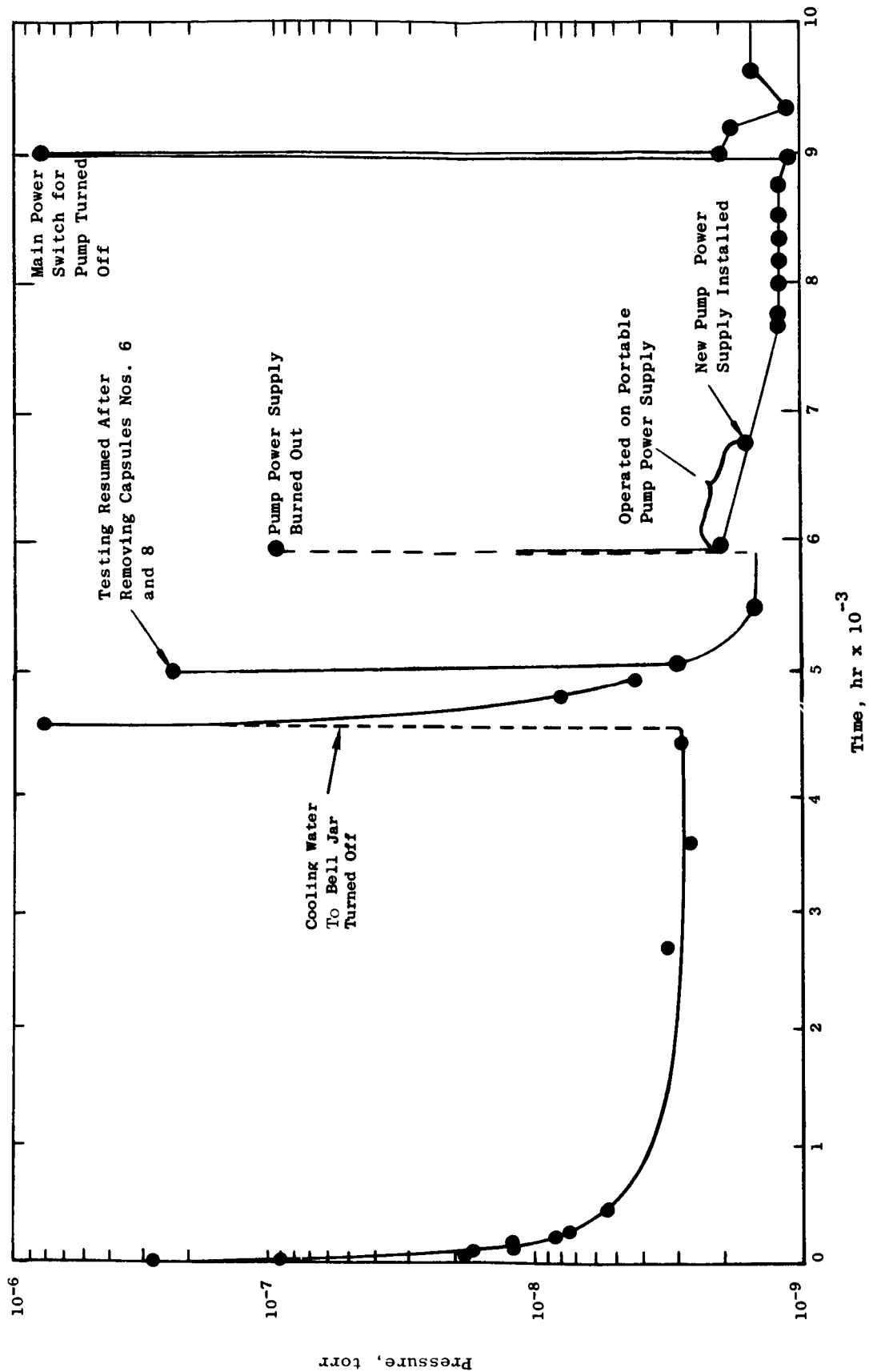


Figure 11. Pressure Profile of High Vacuum Chamber C-III During Testing of Cb-1Zr Alloy Reflux Corrosion Capsule No. 7 and AS-55 Alloy Reflux Corrosion Capsule No. 9 at 2000°F for 10,000 Hr.

a maximum of 4.4×10^{-6} torr. After approximately 3000 hr of testing, the chamber was opened to repair a heater which was shorting to the tantalum shielding. At the end of the 5000-hr exposure, the chamber was opened and capsules No. 12 (AS-55 alloy) and No. 2 (D-43 alloy) were removed from the chamber for evaluation. The results of these 5000-hr tests are also reported in Interim Report 2.² Capsules No. 11 (AS-55 alloy) and No. 4 (D-43 alloy) were put back on test to complete the 10,000 hr of testing. After approximately 7200 hr, the test was shutdown so that the entire test facility could be moved to a new laboratory. Testing was resumed and completed without difficulty; the chamber was not opened at any time during this move. The pressure profile for capsules No. 11 (AS-55 alloy) and No. 4 (D-43 alloy) over the 10,000-hr test period is shown in Figure 12.

Temperature control was accomplished by using a voltage regulated, variac controlled supply to a step-down transformer which fed the power to the tantalum heaters. Two heaters were connected in series so that only two power supplies were required for testing four capsules. The average measured temperatures of capsules No. 7, No. 9, No. 11, and No. 4 during testing are shown in Tables V and VI.

Two commonly used techniques for determining the reflux rate of potassium in capsules are: 1) a direct calculation of the heat radiated from the condensing region of the capsule to the vacuum chamber wall and 2) an indirect calculation of the heat radiated from the condensing region by the subtraction of the heat losses occurring in the capsule, in regions other than the condensing region, from the total heat input to the capsule. Because of the difficulties in determining the heat losses of the capsules in the experimental setup described in this report, the heat radiated from the condensing region of the capsule to the vacuum chamber wall was calculated using the first method cited. The heat flux was calculated using the equation

$$q_{12} = A_1 \sigma \left[\frac{1}{\frac{1}{\epsilon_1} + \frac{A_1}{A_2} \left(\frac{1}{\epsilon_2} - 1 \right)} \right] (T_1^4 - T_2^4) \quad 1$$

Assuming coaxial cylinders and $T_2 = 0^\circ\text{R}$

and letting

A_1	=	0.0327 ft ²	(radiating area of capsules)
A_2	=	7.85 ft ²	(absorbing area of chamber)
ϵ_1	=	0.4	(emissivity of capsule)
ϵ_2	=	0.2	(emissivity of chamber)
T_1	=	2410°R	(temperature of capsule surface)
σ	=	0.174 (10 ⁻⁸) Btu hr ⁻¹ ft ⁻² °F ⁻⁴	(Stefan-Boltzmann's constant)

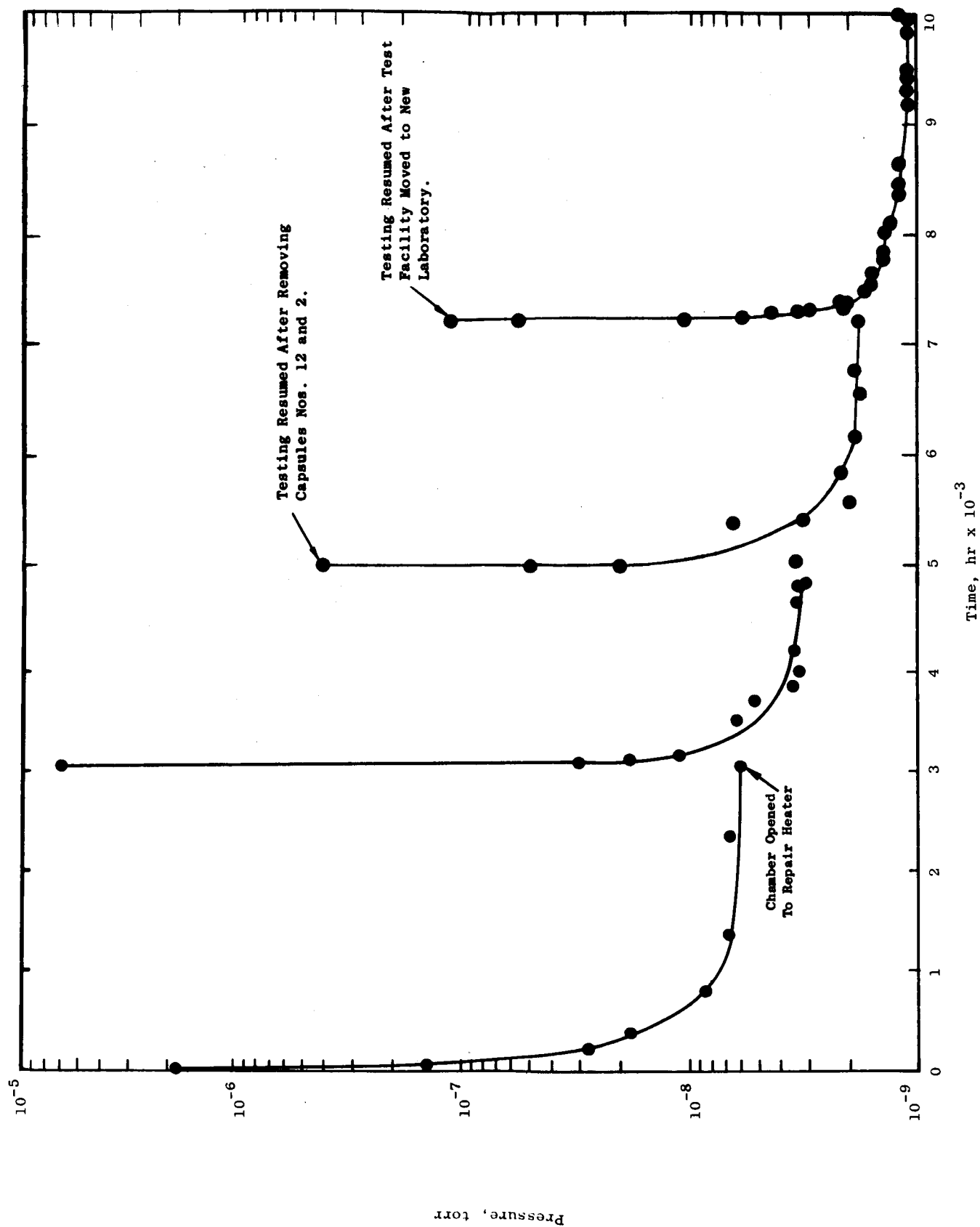


Figure 12. Pressure Profile of High Vacuum Chamber C-II During Testing of AS-55 Alloy Reflux Corrosion Capsule No. 11 and D-43 Alloy Reflux Corrosion Capsule No. 4 at 2000°F for 10,000 Hr.

Table V. Av. Test Temperatures of Reflux Capsule No. 7 (Cb-1Zr Alloy)
And Reflux Capsule No. 9 (AS-55 Alloy) During 10,000-Hr Exposure to Potassium

Capsule	Location of Temp. Measurement	Method of Temp. Measurement	Av. Temp., °F	Mean Deviation, °F
No. 7	Heater Region	Thermocouple	1994	+ 7
	Radiating Region	Thermocouple	1903	+ 21
	Top Region	Brightness Pyrometer ^a	1895	+ 19
		Thermocouple	1899	+ 16
No. 9	Heater Region	Thermocouple	2007	+ 41
	Radiating Region	Thermocouple	1904	+ 16
	Top Region	Brightness Pyrometer ^a	1894	+ 22
		Thermocouple	1955	+ 14

^aUncorrected for Pyrex window and emissivity.

Table VI. Av. Test Temperatures of Reflux Capsule No. 11 (AS-55 Alloy)
And Reflux Capsule No. 4 (D-43 Alloy) During 10,000-Hr Exposure to Potassium

Capsule	Location of Temp. Measurement	Method of Temp. Measurement	Av. Temp., Of	Mean Deviation, Of
No. 11	Heater Region	Thermocouple	1982	± 27
	Radiating Region	Thermocouple	^a ---	^a ---
		Brightness Pyrometer ^b	1913	± 12
	Top Region	Thermocouple	1920	± 17
No. 4	Heater Region	Thermocouple	2055	± 34
	Radiating Region	Thermocouple	^a ---	^a ---
		Brightness Pyrometer ^b	1927	± 19
	Top Region	Thermocouple	1845	± 26

^aNo reading; thermocouple failed.

^bUncorrected for Pyrex window and emissivity.

then,

$$q_{12} = 762 \text{ Btu/hr}$$

The refluxing rate w (lb/hr/ft²) is calculated from the equation

$$w = \frac{q_{12}}{H_v A_3} \quad 2$$

where $H_v = 745 \text{ Btu/lb}$ for potassium at 1950°F (reference 8)
 $A_3 = 0.0275 \text{ ft}^2$ (area of condensing surface)

Then,

$$w = 37.2 \text{ lbs/hr/ft}^2$$

The reflux rate, however, can vary significantly with variations in the measured and assumed test conditions. By letting the emittance of the radiating surface ϵ_1 vary from 0.3 to 0.5, the emissivity of the chamber wall ϵ_2 vary from 0.1 to 0.3, and the temperature of the radiating surface vary from 1900°F to 2000°F, it is possible for the reflux rate to vary from a minimum 25.2 lb/hr/ft² to a maximum 51.1 lb/hr/ft². Further, if it is assumed that half the radiating surface of the capsule is exposed to a heat sink that is at a temperature of approximately 1000°F, which is probably more realistic in the case of the test setup for these capsules, similar calculations using equations 1 and 2 show the possible range for the reflux rate to be 23.4 lb/hr/ft² to 48.0 lb/hr/ft². Overall, the refluxing rate probably was approximately $37 \pm 12 \text{ lb/hr/ft}^2$.

It should be noted that, in more recent work at General Electric under NASA sponsorship,⁵ a facility has been built and operated which provides a more accurate determination of the heat flux from the condensing region of the capsule than either of the previously discussed techniques. This facility utilizes a heat exchanger around the condensing region of the capsule. The heat from the capsule is radiated to the heat exchanger and is transferred to a flowing water system, allowing an accurate heat balance to be performed on the water system.

Results of 10,000-Hr Reflux Capsule Corrosion Tests

One Cb-1Zr alloy capsule (No. 7), two AS-55 alloy capsules (Nos. 9 and 11), and one D-43 alloy capsule (No. 4), containing purified potassium, completed the 10,000 hr of testing at 2000°F under refluxing conditions. Visual examination of the Cb-1Zr alloy capsule No. 7 revealed discolorations in the region between the primary condensing zone and the liquid zone (Figure 13). Also found in this region were black deposits with a gold discoloration of the metal around or near the deposits (Figure 14).

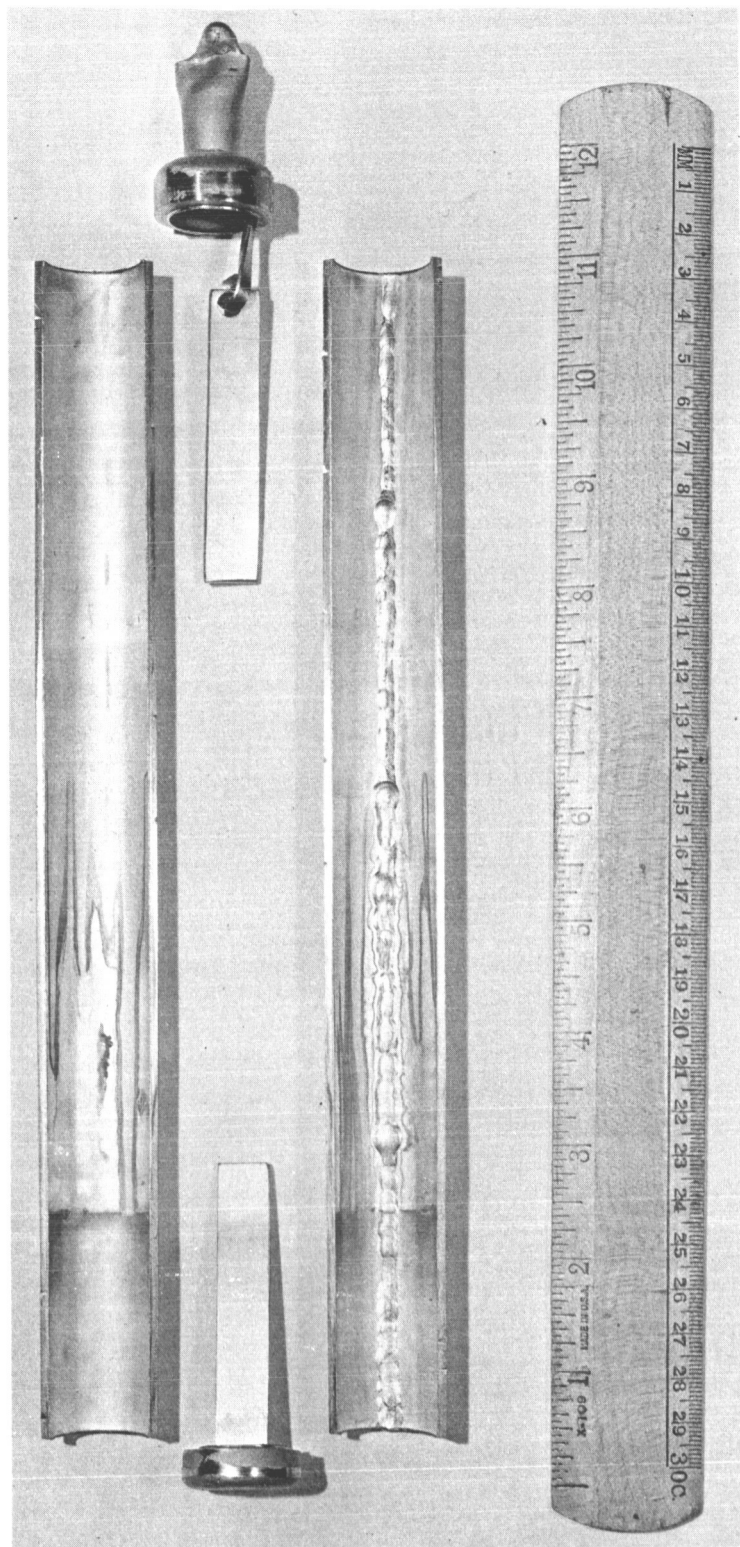


Figure 13. Sectioned Cb-1Zr Alloy Reflux Corrosion Capsule No. 7 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

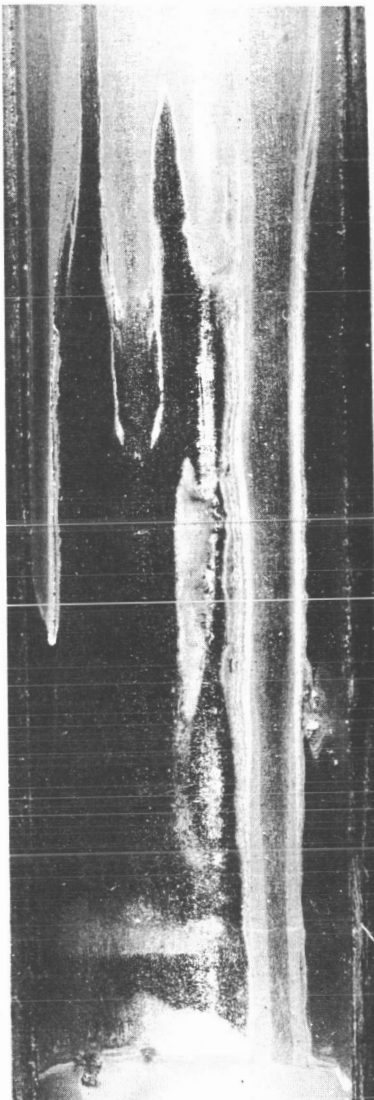


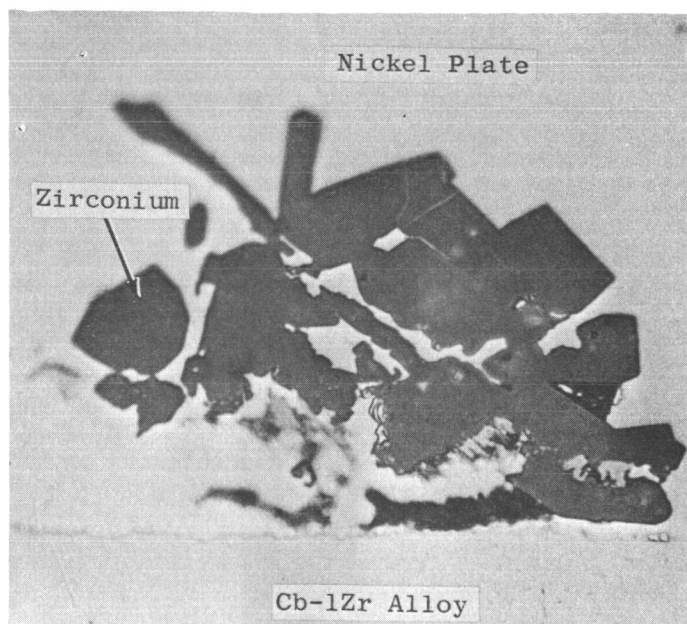
Figure 14. Black Deposits and Discoloration Observed on Inner Surface of Cb-1Zr Alloy Corrosion Capsule No. 7 Between Primary Condensing Zone and Liquid Zone After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

Mag.: 2 X

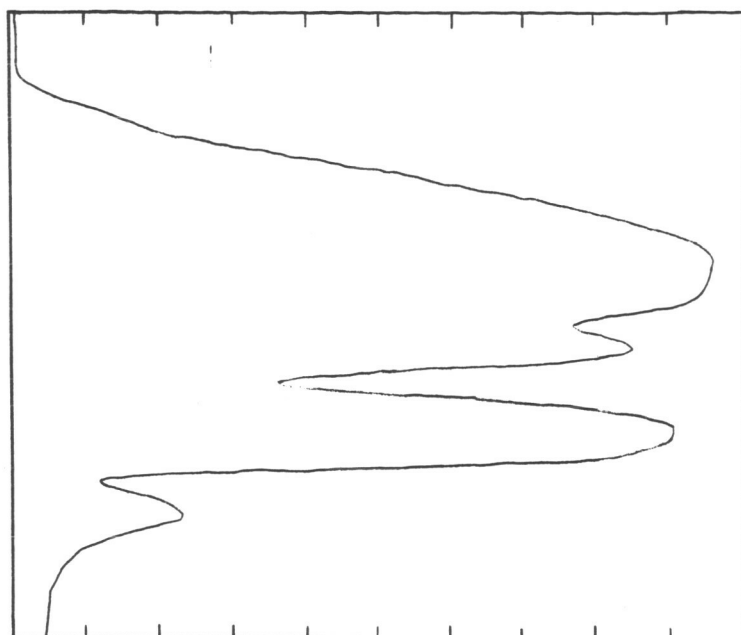
In addition, a change in the general surface appearance of the heat-affected zone of the weld and a more lustrous appearance in the surface above the liquid zone was noted.

Metallographic examination of that region where the deposits were observed revealed a buildup of one phase to a thickness as extensive as 0.002 in.; a second phase also observed was considerably thinner and less abundant (Figure 15). Electron microprobe analyses revealed that both phases had the same composition, i.e., essentially all zirconium (Figure 15) with a small amount of columbium. The low zirconium content observed in Figure 15.b results from the fact that the area of the electron beam is larger than the deposit being analyzed; when an appropriate area correction is applied, the deposit is essentially all zirconium. Oxygen, nitrogen, or carbon were not detected. Note, however, that the detectable limits for oxygen, nitrogen, and carbon using the electron microprobe analyzer are somewhat questionable. Although detection of these elements above one to two wt % has been achieved at this laboratory, detection below one to two wt %, especially for oxygen and nitrogen, is considered very doubtful. The larger and more abundant dark grey phase is believed to be α -zirconium; the less abundant phase, retained β -zirconium. Also, significantly, the electron microprobe analyses revealed a zirconium content of approximately 5% in the capsule wall adjacent to the deposits compared with a zirconium content of 1.3% as determined by chemical analysis of the original sheet before test and by electron microprobe analyses of the capsule OD in the condensing zone after the test exposure. As discussed on page 52, metallographic examination of the capsule inside diameter surface suggests that the zirconium observed in the deposits was leached from the condensing zone. It should be noted that the presence of zirconium deposits is not a general condition inasmuch as they were not found in the AS-55 and D-43 capsules, suggesting that either these alloys behave differently or that the potassium purity influences the zirconium transfer. Both D-43 capsules and the AS-55 capsule No. 11 were filled with potassium using the improved vacuum filling facility and, as will be discussed, the yttrium, rather than the zirconium, in AS-55 would appear to dominate any interactions with oxygen contained as an impurity in the potassium.

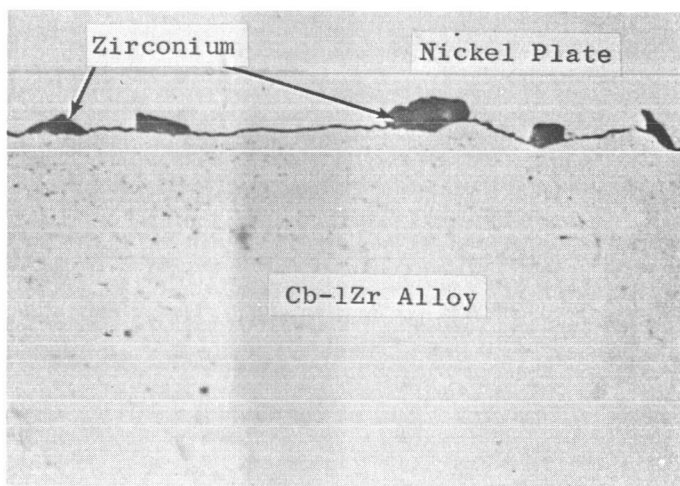
The AS-55 alloy capsule No. 9 also showed discoloration, to a much lesser degree than those found in Cb-1Zr alloy capsule No. 7, in the region between the primary condensing zone and the liquid zone (Figure 16). A dark grey spot surrounded by a white deposit, similar to those observed in AS-55 alloy capsule No. 12 which was tested 5000 hr, was noted on the inside wall in the liquid region opposite the location where a thermocouple was attached to the outer wall. Metallographic examination of this region revealed a 0.0002-in. thick buildup which is suspected to be Y_2O_3 (Figure 17). Also, as in capsule No. 7 (Cb-1Zr alloy), a change in the general surface appearance



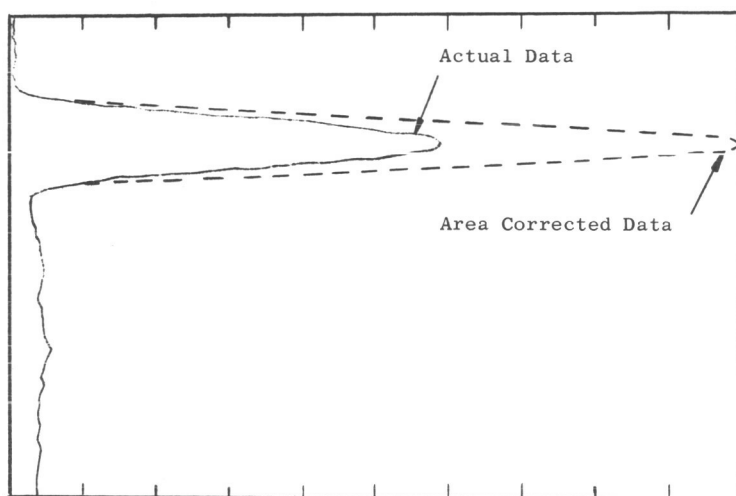
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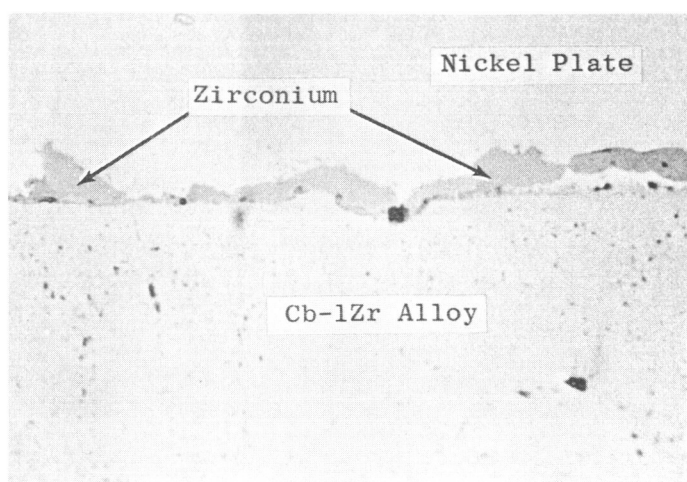
c.



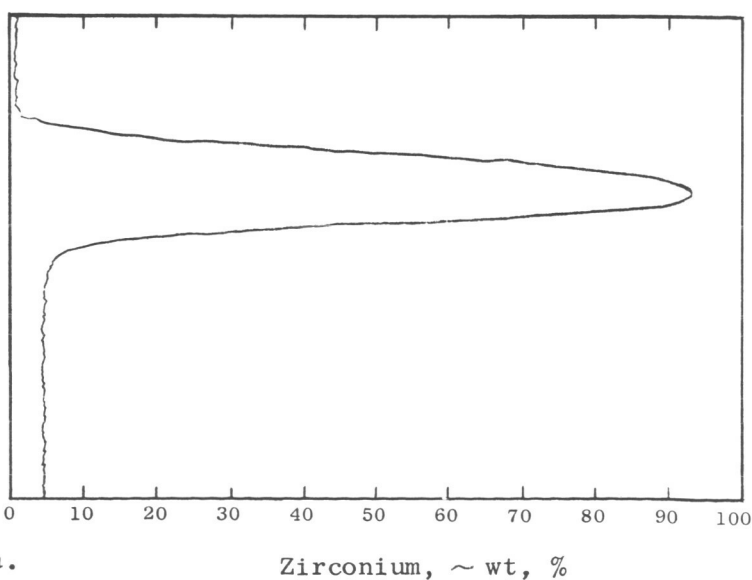
(A010114)



b.



(A010113)



a.

Etchant: None

Mag.: 1000X
N.A.: 0.85

Zirconium, ~ wt, %

Figure 15. Photomicrographs and Electron Microprobe Analyses of Deposits Observed in Region Between Primary Condensing Zone and Liquid Zone of Cb-1Zr Alloy Reflux Corrosion Capsule No. 7 After 10,000-Hr Exposure to Potassium at 2000°F in Vacuum of 10^{-9} Torr.

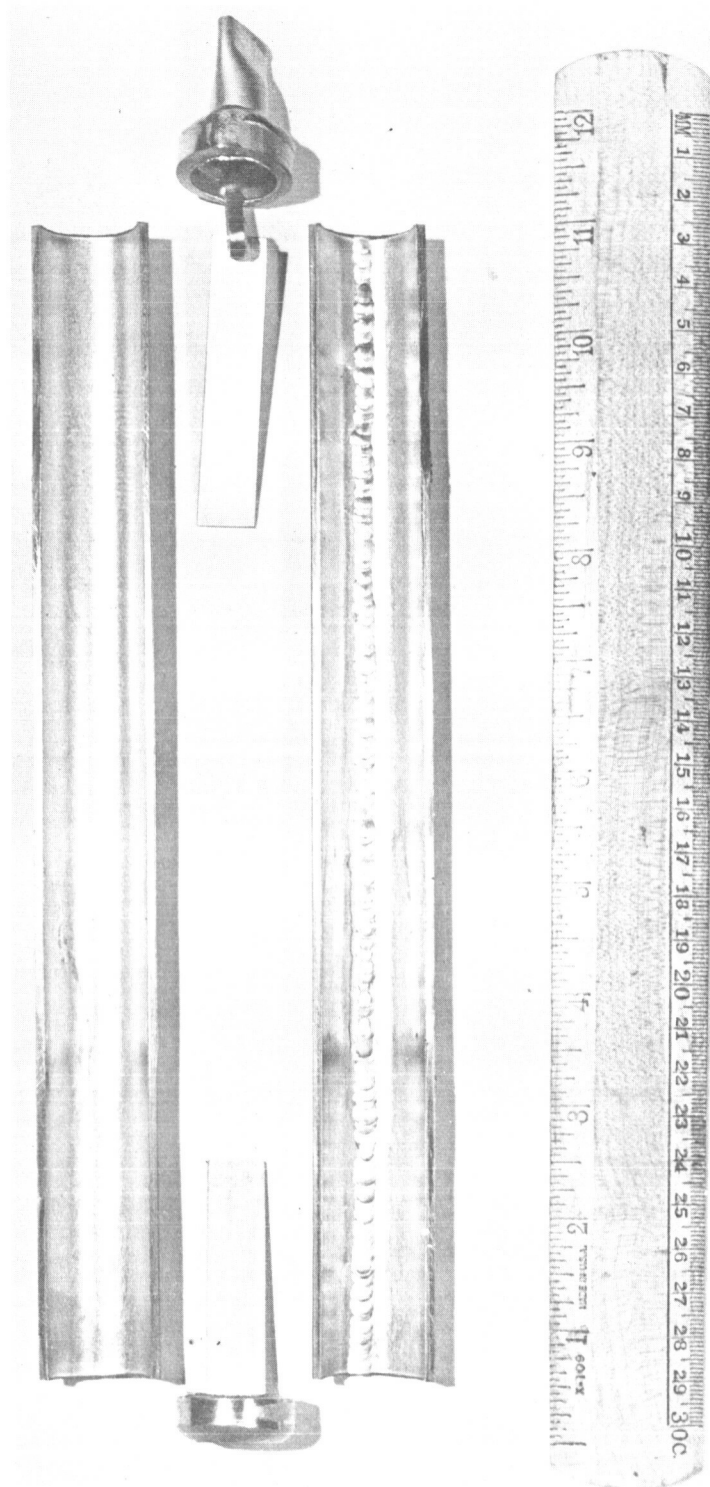
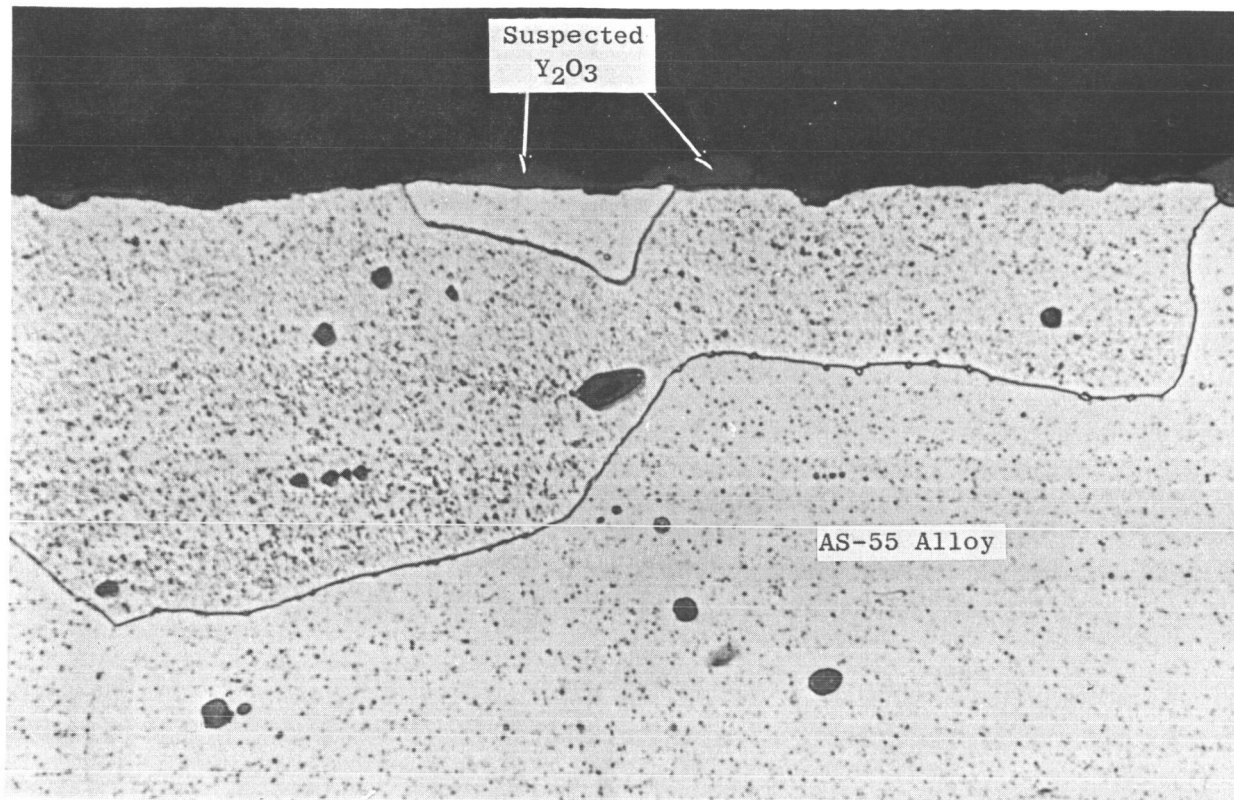


Figure 16. Sectioned AS-55 Alloy Reflux Corrosion Capsule No. 9 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.



(K809)

Figure 17. Deposit on ID of AS-55 Alloy Reflux Corrosion Capsule No. 9 in Liquid Zone Opposite Thermocouple Location on Outer Wall After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

in the heat-affected zone of the weld and a more lustrous appearance above the liquid zone were observed.

AS-55 alloy capsule No. 11, which was filled with potassium using the vacuum filling facility discussed earlier, showed no discolorations in the region just above the liquid zone (Figure 18). White deposits, however, also similar to those seen previously in the 5000-hr AS-55 alloy capsule No. 12, were found in three locations on the inside surface of AS-55 capsule No. 11: two in the liquid region; one in the vapor region. In the previous capsules, viz., 5000-hr tests, the white deposits were found in the liquid zone only. Figure 19 shows the white deposits that formed on the weld in the liquid zone; Figure 20, a deposit on the parent metal in the liquid region opposite the point where a thermocouple was in contact with the capsule wall. Although most of the deposited material was bonded loosely to the capsule surface and tended to spall, metallographic examination of the weld in the liquid zone revealed some deposited material that apparently was more adherent (Figure 21).

Yttrium was the only metal detected by spectrographic analyses of the white deposits in the AS-55 alloy capsules. Similarly, electron microprobe analyses of the deposits observed metallographically confirmed that yttrium was the only major constituent. X-ray diffraction studies of the deposits were unsuccessful in identifying the structure. Therefore, it is postulated that the deposit is Y_2O_3 formed by the oxidation of yttrium in the potassium and on the surface of the capsule wall. The primary mechanism believed to account for the presence of Y_2O_3 at localized areas on the capsule wall assumes the existence of localized areas in the capsule wall which can provide a relatively large source of oxygen and effect the oxidation of yttrium that is in solution with the potassium at these specific locations. In the case of the weld metal, a high oxygen concentration could exist because of contamination during the welding operation. The presence of high oxygen concentration in localized spots in the parent metal opposite thermocouple junctions is most likely because of diffusion of oxygen from the thermocouple insulation resulting from nonstoichiometry, entrapment of air during swagging, or from impurities in the Al_2O_3 . The latter possibilities recommend the use of bare thermocouple wires.

A second mechanism that could play a part in the formation of Y_2O_3 at localized areas is the mass transport of yttrium caused by a small localized temperature gradient and the subsequent oxidation of the yttrium to Y_2O_3 by reaction with oxygen in the potassium and/or in the capsule material. Since the location of the deposits either was at the bottom corner of the capsules or corresponded to the location where the tantalum-sheathed thermocouples had bonded to the outer capsule wall, heat could possibly be extracted from the capsule wall at these locations by conduction through the capsule support at the bottom and by conduction along the tantalum sheath to result in "cold spots".

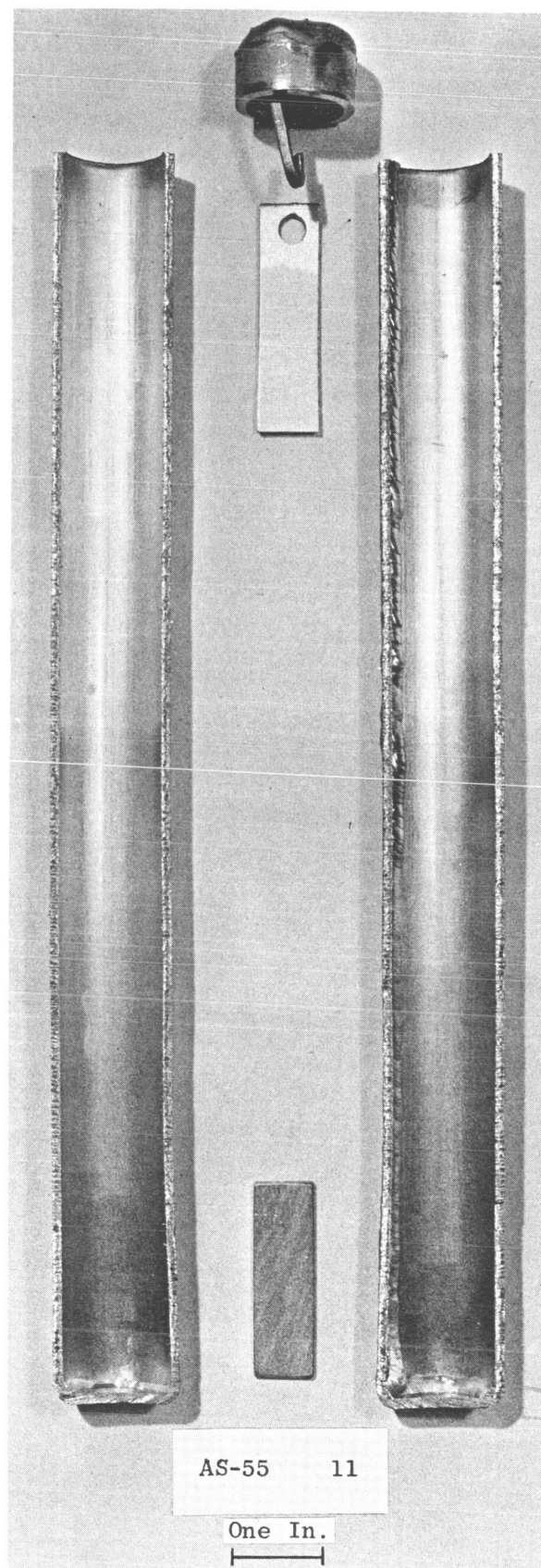


Figure 18. Sectioned AS-55 Alloy Reflux Corrosion Capsule No. 11 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

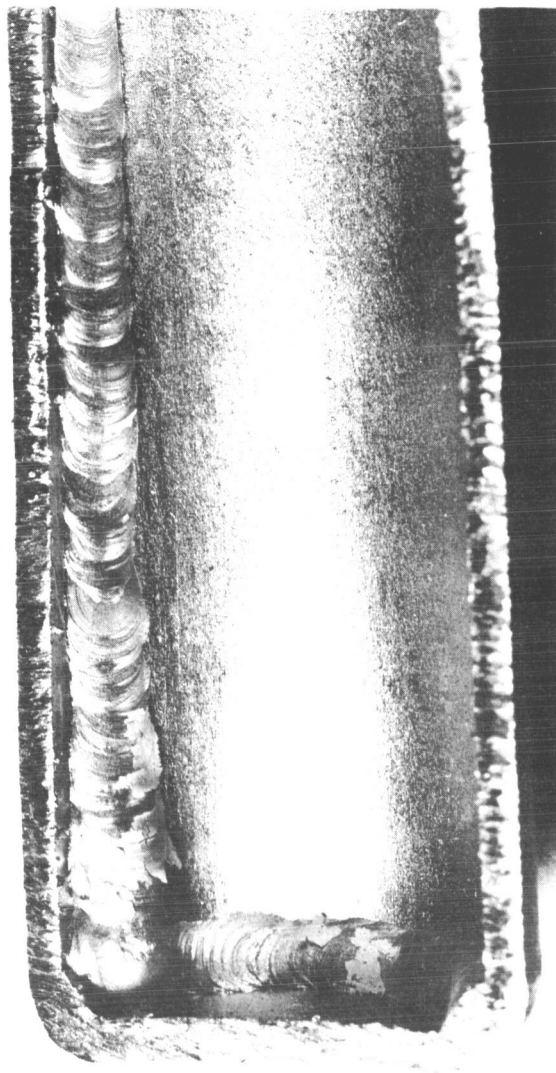


Figure 19. White Deposit Observed in Weld in Liquid Zone of AS-55 Alloy Reflux Capsule No. 11 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

Mag.: 3X

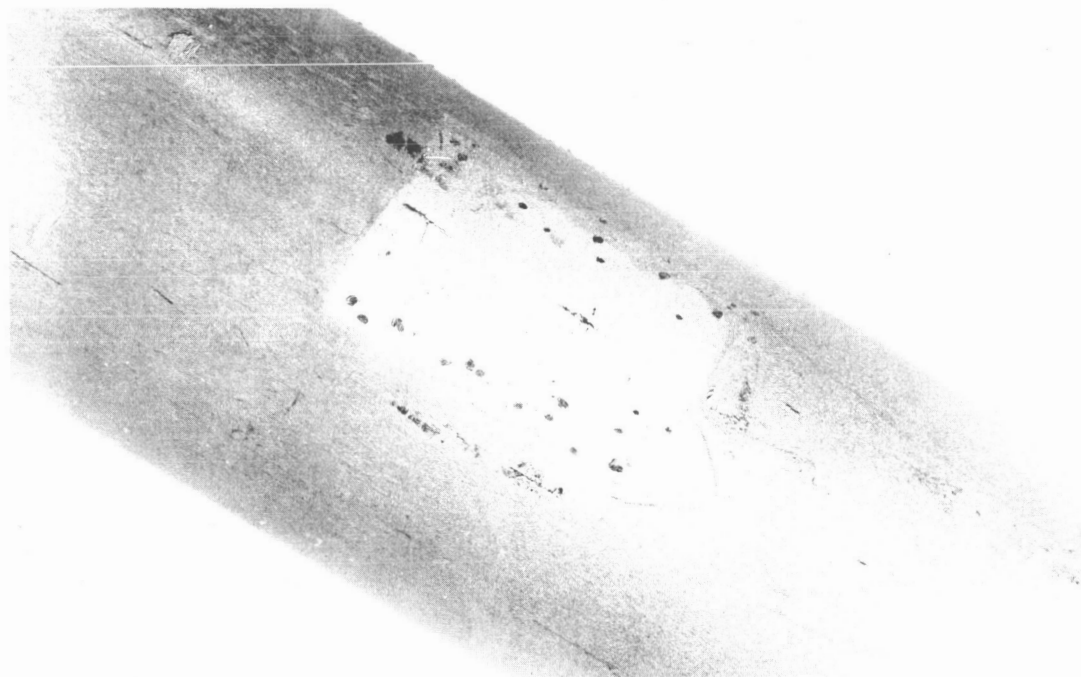
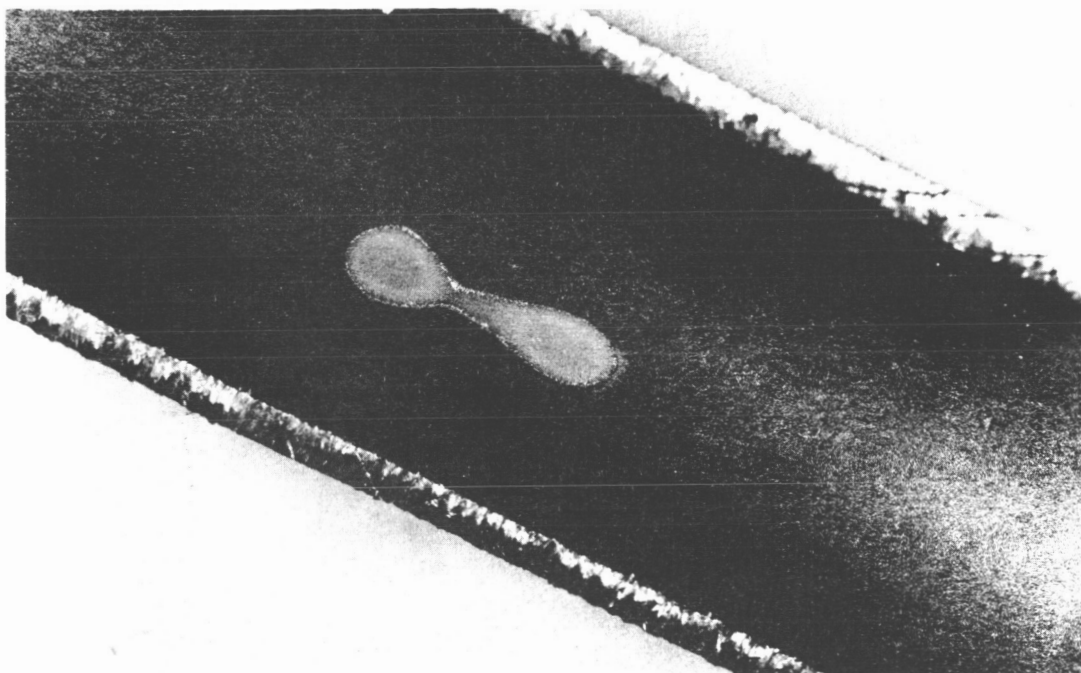
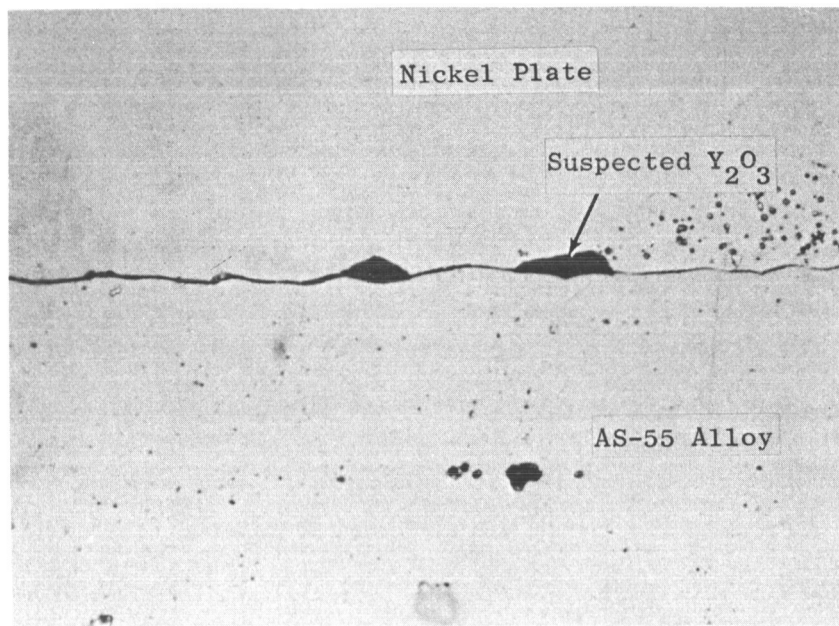


Figure 20. White Deposits Observed on Inside Surface of AS-55 Alloy Reflux Capsule No. 11 in Liquid Zone (Top) Opposite Thermocouple Location on the Capsule Wall (Bottom); Capsule Exposed to Potassium for 10,000-Hr at 2000°F in a Vacuum of 10^{-9} Torr.

Mag.: 3X



(A160211)

Figure 21. Adherent White Deposit Observed on Weld in Liquid Zone of AS-55 Alloy Reflux Corrosion Capsule No. 11 After 10,000-Hr Exposure to Potassium at 2000^oF in a Vacuum of 10⁻⁹ Torr.

Etchant: None

Mag.: 1000X

N.A.: 0.85

Both mechanisms cited assume some solubility of yttrium in potassium. As discussed previously, although the solution of yttrium in potassium has not been observed experimentally, yttrium has been demonstrated soluble to some extent in a lithium-columbium system at 1500°F.⁹

After sectioning, the D-43 alloy capsule No. 4 revealed no deposits and a lustrous appearance throughout. The liquid-vapor interface and the vapor-condenser interface (Figure 22) were more discernible than those observed in the 5000-hr D-43 alloy capsule No. 2.

In addition to visual examination, the following tests were conducted on specimens machined from the capsule wall (Figure 23) to evaluate the effect of the 10,000-hr exposure to the potassium and to the ultra-high vacuum on the capsule material:

1. Chemical analyses.
2. Metallographic examination.
3. Hardness measurements.
4. Weight and thickness measurements.
5. Bend tests on samples contained within the capsule.
6. Stress-rupture tests at 2000°F on specimens machined from the AS-55 alloy and D-43 alloy capsule walls.

Chemical Analyses. Results of chemical analyses of samples taken at locations shown in Figure 23 are recorded in Table VII. In an attempt to determine the gradient in chemical composition from the internal surface to the external surface, three 0.080-in. thick specimens were taken from the walls of the capsules at each location. The outer two thirds of material was removed from one specimen; the inner two thirds, from the second specimen; the inner and outer one third, from the third specimen. The remaining material was analyzed for oxygen, nitrogen, hydrogen, and carbon.

Differences observed in the nitrogen and hydrogen contents of any of the capsules are not considered significant; variations are attributed to inhomogeneity in the sheet and inaccuracies in the analyses. Similarly, no significance is attached to changes observed in the carbon content of capsules Nos. 4 (D-43 alloy), 11 (AS-55 alloy), and 9 (AS-55 alloy). The increase in carbon content observed in the inside sample from the vapor zone of capsule No. 7 (Cb-12Zr alloy) may result from the liquid potassium, high in impurities near the liquid-vapor interface, splashing upon the capsule wall and being gettered by the zirconium deposit detected visually and metallographically on the inside diameter of that region. Other changes in carbon content observed in capsule No. 7 are insignificant. No mass transfer of carbon was evident in any of the capsules.

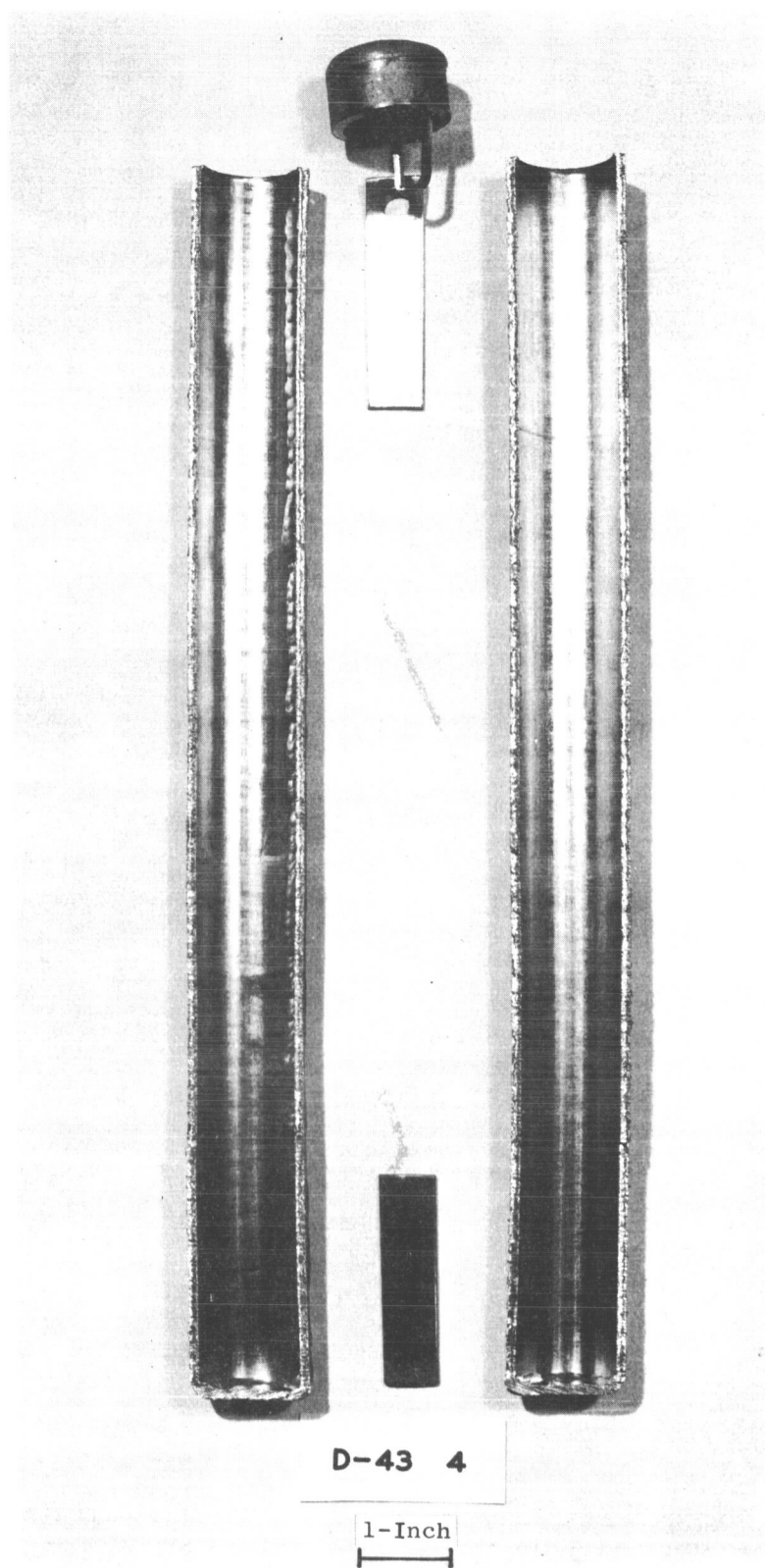


Figure 22. Sectioned D-43 Alloy Reflux Corrosion Capsule No. 4 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

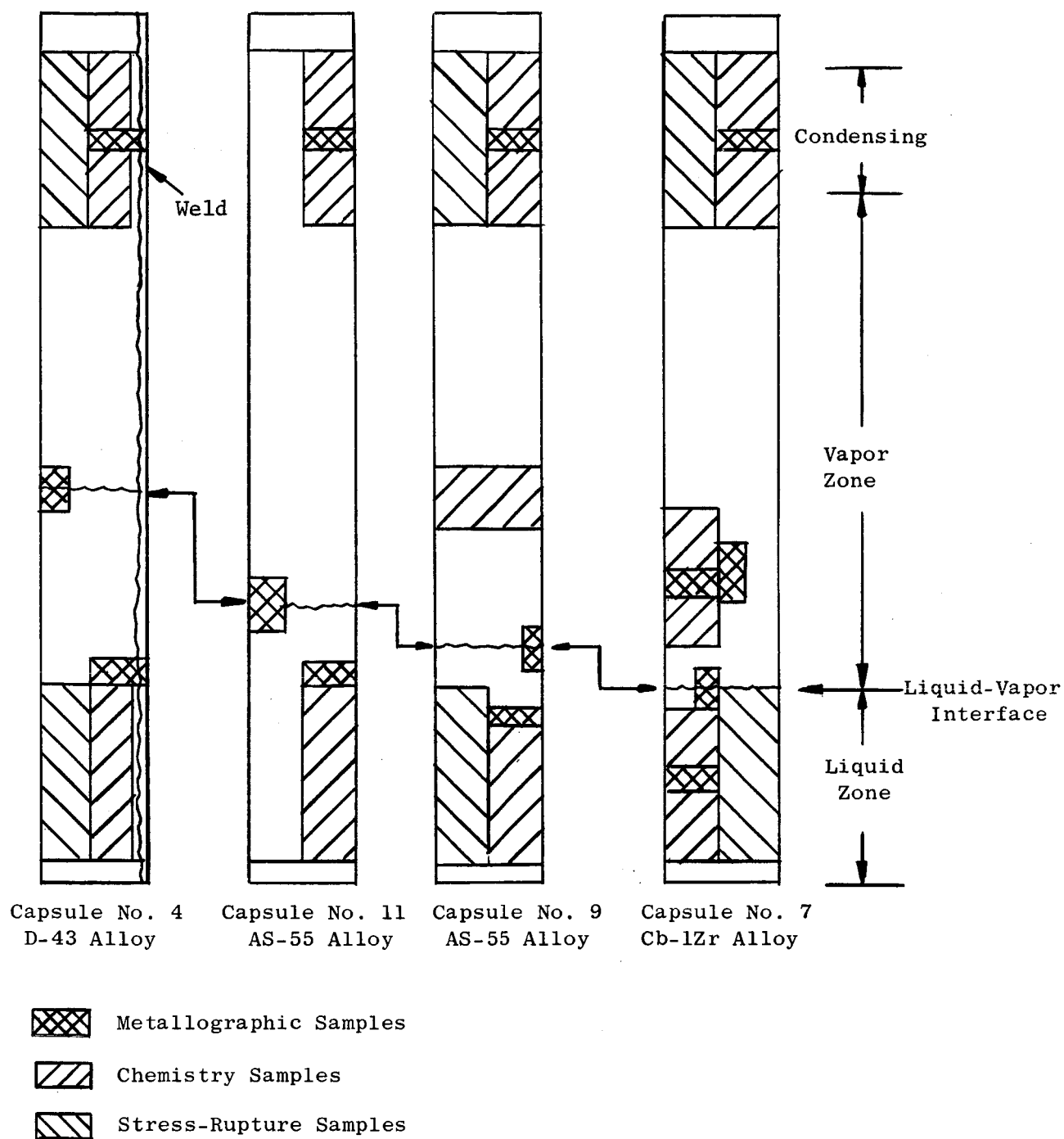


Figure 23. Location of Samples from Wall of Reflux Corrosion Capsules For Chemical Analyses, Metallographic Examination, and Stress-Rupture Tests.

Table VII. Chemical Analyses (Wt. %) of Reflux Capsules After 10,000-Hr Exposure to Potassium at 2000°F

Element	Before Exposure			Chemical Analyses, wt. %						Vapor Zone			Condensing Zone		
	Inside ^a	Center ^b	Outside ^c	Inside ^a	Center ^b	Outside ^{c,h}	Inside ^a	Center ^b	Outside ^{c,h}	Inside ^a	Center ^b	Outside ^{c,h}	Inside ^a	Center ^b	Outside ^{c,h}
Capsule No. 4 (D-43, DuPont 322) ^g															
Carbon	0.090 ^d	0.111 ^d	0.103 ^d	0.0870 ^d	0.0670 ^d	0.0770 ^d	---	---	---	0.0310 ^d	0.0920 ^d	---	0.0310 ^d	0.0920 ^d	0.1000 ^d
Oxygen	0.0120 ^d	0.0106 ^d	0.0105 ^d	0.0241	0.0252	0.0342	---	---	---	0.0143	0.0125	---	0.0143	0.0125	0.0160
Nitrogen	0.0023 ^d	0.0025 ^d	0.0026 ^d	0.0045	0.0041	0.0042	---	---	---	0.0031	0.0029	---	0.0031	0.0029	0.0037
Hydrogen	0.0002	0.0008	0.0003 ^d	0.0002	0.0012	0.0012	---	---	---	0.0001	0.0001	---	0.0001	0.0001	0.0003
Capsule No. 11 (AS-55, NAS-5515) ^g															
Carbon	0.104 ^d	0.071 ^d	0.145 ^d	0.0630 ^d	0.0670 ^d	0.0620 ^d	---	---	---	0.0770 ^d	0.0750 ^d	---	0.0770 ^d	0.0750 ^d	0.0670 ^d
Oxygen	0.0403 ^d	0.0240 ^d	0.0424 ^d	0.0137 ^d	0.0162 ^d	0.0443	0.0226 ^d	---	---	0.0263 ^d	0.0206 ^d	---	0.0263 ^d	0.0206 ^d	0.0672
Nitrogen	0.0203 ^d	0.0184 ^d	0.0189 ^d	0.0183 ^d	0.0165 ^d	0.0192	0.0146 ^d	---	---	0.0173 ^d	0.0216 ^d	---	0.0173 ^d	0.0216 ^d	0.0192
Hydrogen	0.0002 ^d	0.0002 ^d	0.0001	0.0003	0.0002	0.0001	0.0006 ^d	---	---	0.0002 ^d	0.0002 ^d	---	0.0002 ^d	0.0002 ^d	0.0003
Capsule No. 9 (AS-55, NAS-555) ^f															
Carbon	---	---	---	0.0670	0.0670 ^d	0.0670	0.0740	0.0680 ^d	0.0660 ^d	0.0710 ^d	0.0660 ^d	0.0660 ^d	0.0710 ^d	0.0660 ^d	0.0730 ^d
Oxygen	---	---	---	0.0304 ^e	0.0173 ^e	0.0502 ^d	0.0254 ^d	0.0212 ^d	0.0381 ^d	0.0260 ^d	0.0212 ^d	0.0381 ^d	0.0260 ^d	0.0435 ^e	0.0610 ^d
Nitrogen	---	---	---	0.0203 ^e	0.0203 ^e	0.0227 ^d	0.0213 ^d	0.0206 ^d	0.0215 ^d	0.0198 ^e	0.0206 ^d	0.0215 ^d	0.0198 ^e	0.0177 ^e	0.0179 ^d
Hydrogen	---	---	---	0.0006 ^e	0.0003 ^e	0.0005 ^d	0.0002	0.0002 ^d	0.0003 ^d	0.0004 ^e	0.0002 ^d	0.0003 ^d	0.0004 ^e	0.0005 ^e	0.0003 ^d
Capsule No. 7 (Cb-12r, Stellite-519) ^f															
Carbon	0.006 ^d	0.004 ^d	0.007 ^d	0.0060	0.0060 ^d	0.0080 ^d	0.0170	0.0080 ^d	0.0090 ^d	0.0060 ^d	0.0080 ^d	0.0090 ^d	0.0060 ^d	0.0090 ^d	0.0110
Oxygen	0.0134 ^d	0.0087 ^d	0.0095 ^d	0.0208 ^d	0.0120 ^d	0.0251 ^d	0.0862 ^d	0.0211 ^d	0.0318 ^d	0.0079 ^d	0.0211 ^d	0.0318 ^d	0.0079 ^d	0.0073 ^d	0.0178 ^d
Nitrogen	0.0047 ^d	0.0047 ^d	0.0048 ^d	0.0066 ^d	0.0047 ^d	0.0035 ^d	0.0044 ^d	0.0053 ^d	0.0032 ^d	0.0052 ^d	0.0053 ^d	0.0032 ^d	0.0052 ^d	0.0055 ^d	0.0050 ^d
Hydrogen	0.0002 ^d	0.0002 ^d	0.0002 ^d	0.0003	0.0004 ^d	0.0003 ^d	0.0007 ^d	0.0010 ^d	0.0005 ^d	0.0003 ^d	0.0010 ^d	0.0005 ^d	0.0003 ^d	0.0004 ^d	0.0003 ^d

^a Inside 1/3 of capsule wall.

^b Center 1/3 of capsule wall.

^c Outside 1/3 of capsule wall.

^d Average of two analyses; gas analyses by vacuum fusion techniques.

^e Average of three analyses; gas analyses by vacuum fusion techniques.

^f High oxygen potassium.

^g Low oxygen potassium.

^h Outside surface grit blasted with -280 mesh aluminum powder before test.

The oxygen concentration in the outer wall portions of essentially all the capsules increased. The increase in oxygen in the outside specimens is accredited to the fact that the outer surface of the capsules were grit blasted with Al_2O_3 . Similar increases were observed in the outer surfaces of capsules exposed for 5000 hr. More recent work performed at this laboratory has demonstrated that the apparent oxygen contamination from grit blasting with Al_2O_3 is severe; a 0.0175-in. thick sheet of Cb-1Zr alloy exhibited an oxygen increase of 842 ppm after grit blasting with Al_2O_3 .⁹

A comparison of the oxygen analyses from the inner wall specimens from Cb-1Zr alloy capsules Nos. 6 (exposed 5000 hr) and 7 (exposed 10,000 hr), indicated that 1) the oxygen content in the condensing zone of capsule No. 7 was significantly lower than that of capsule No. 6 2) the oxygen content in the vapor zone of capsule No. 7 was considerably larger than that of capsule No. 6. Essentially no difference was observed in the oxygen contents in the liquid zone. These data are illustrated in Figure 24. An examination of the oxygen analysis of the inner portion of the capsule wall before the 10,000-hr exposure to potassium suggests that leaching of oxygen by the high purity condensate occurred in the condensing zone of capsule No. 7. The high oxygen concentration in the vapor zone of the Cb-1Zr alloy capsule No. 7 may be the result of a gettering action of the zirconium deposits observed visually and metallographically on the inside diameter of that region. The oxygen could be supplied by the condensate, now rich in oxygen from the condensing zone, and/or the liquid potassium, high in impurities near the liquid-vapor interface, washing upon the capsule wall in that region. Since the changes in oxygen content observed in capsule No. 6 after a 5000-hr exposure appear to be amplified in the 10,000-hr exposure (capsule No. 7), some leaching of oxygen from the condensing zone possibly could have occurred in Cb-1Zr alloy capsule No. 6 and not been recognized because the reported oxygen content of the Cb-1Zr alloy before exposure may be slightly lower than the actual oxygen content. In any case, the transfer of oxygen during the 5000-hr testing was very small. Also, any gettering action of the Cb-1Zr alloy in the liquid zone in both the 5000-hr capsule (No. 6) and 10,000-hr capsule (No. 7) would appear to be relatively small. In view of the leaching in the condensing zone more pronounced evidence of increased oxygen throughout the liquid zone would ordinarily be expected. As indicated, however, this possibly was not observed because oxygen was getterred extensively by the zirconium deposits in the vapor zone.

The oxygen analyses obtained from the inner wall specimens of AS-55 alloy capsule No. 9 revealed a higher oxygen content in the liquid than in the condensing zone. These results agree with the oxygen analyses obtained for AS-55 alloy capsules Nos. 8 and 12 which were exposed for 5000 hr. Except for the analysis from the liquid zone, the oxygen analyses obtained from the inner wall specimens of AS-55 alloy capsule No. 11 agree with the data for the other AS-55 alloy capsules (Nos. 8, 9, and 12). The oxygen content in the inner wall

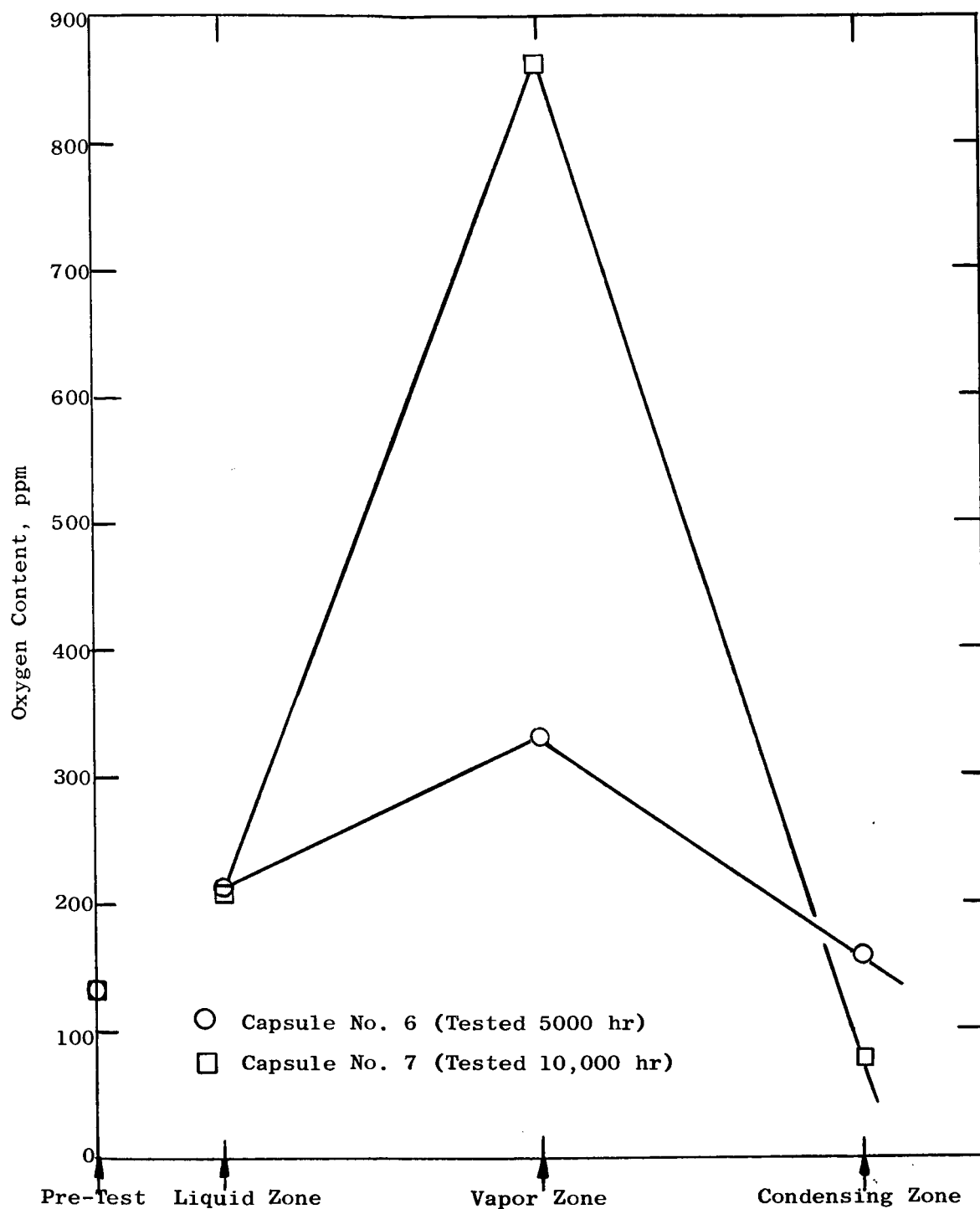


Figure 24. Oxygen Content of Inner 0.027-In. Layer of Cb-1Zr Alloy Capsule Wall as a Function of Specimen Location (Capsules Exposed to Potassium at 2000°F in a Vacuum of 10^{-9} Torr).

sample from the liquid zone of AS-55 alloy capsule No. 11 was considerably lower than that in the other AS-55 alloy capsules. Pertinent data are illustrated in Figure 25.

In comparing the oxygen analyses of AS-55 alloy capsules Nos. 8, 9, 11, and 12 with the analyses before exposure, a loss of oxygen in the condensing zone is apparent and, most likely, was the result of leaching by the high purity condensate. No large differences in the condensing zone are apparent between the 5000- and 10,000-hr tests. Examination of the oxygen contents in the liquid zone reveals that the oxygen leached from the condensing zone remained in the liquid potassium, evidenced by the fact that the oxygen was not gettered by the yttrium-zirconium in the AS-55 alloy capsule wall. It may be hypothesized that the oxygen reacted with yttrium/zirconium taken in solution in the liquid potassium to form finely divided Y_2O_3 or a complex yttrium-zirconium oxide. Although the solution of yttrium in potassium has not been observed experimentally, as related previously, yttrium has been demonstrated to be soluble, to some extent, in a lithium-columbium system at $1500^\circ F$.⁸ Also, it is possible for the oxygen to react with yttrium/zirconium at the surface of the capsule wall in locations other than that which was chemically analyzed. The substantial loss of oxygen observed in the liquid zone of AS-55 capsule No. 11 may be the result of similar phenomena and/or the fact that the potassium in capsule No. 11 was of higher purity than that in capsule No. 9. It should be noted that AS-55 alloy capsule No. 11, which exhibited the extensive loss of oxygen in the liquid zone, contained the largest amount of the white deposit visually observed in any of the AS-55 alloy capsules. This white deposit (discussed earlier) is presumed to be Y_2O_3 .

A comparison of the oxygen content of the inner wall specimens from D-43 alloy capsule No. 4 with the analyses before exposure reveals no apparent leaching of oxygen from the condensing zone and a definite gettering of oxygen in the liquid zone. Little difference in the oxygen content in the condensing zone is observed between D-43 alloy capsule No. 2 exposed for 5000 hr, and D-43 alloy capsule No. 4, exposed for 10,000 hr. Considerably more gettering of oxygen in the liquid zone is evident in the longer test (D-43 alloy capsule No. 4). These data are illustrated in Figure 26. It should be noted that the suspected difference in the purity of the potassium used to fill capsules Nos. 6 and 7 (Cb-1Zr alloy) and that used to fill capsules Nos. 2 and 4 (D-43 alloy) does not seem to affect the magnitude of the oxygen gradient observed between the condensing zone and the liquid zone of the capsule after the test exposure.

Electron microprobe analyses disclosed no loss of zirconium from the outside diameters of the Cb-1Zr alloy and D-43 alloy capsules tested for 10,000 hr. Nor was a loss of zirconium or yttrium found in the AS-55 alloy capsules tested for 10,000 hr.

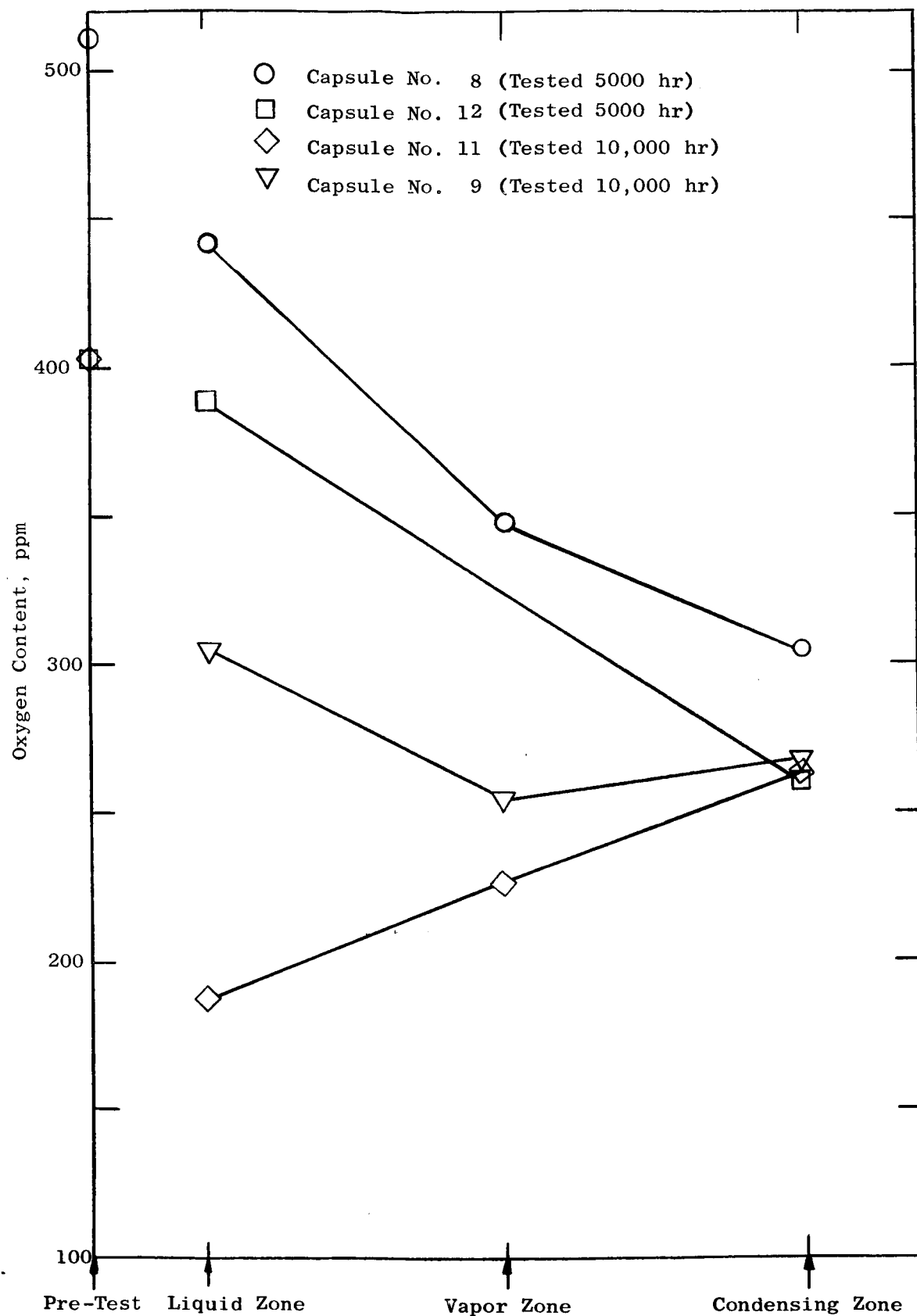


Figure 25. Oxygen Content of Inner 0.027-In. Layer of AS-55 Alloy Capsule Wall as a Function of Specimen Location (Capsules Exposed to Potassium at 2000°F in a Vacuum of 10^{-9} Torr).

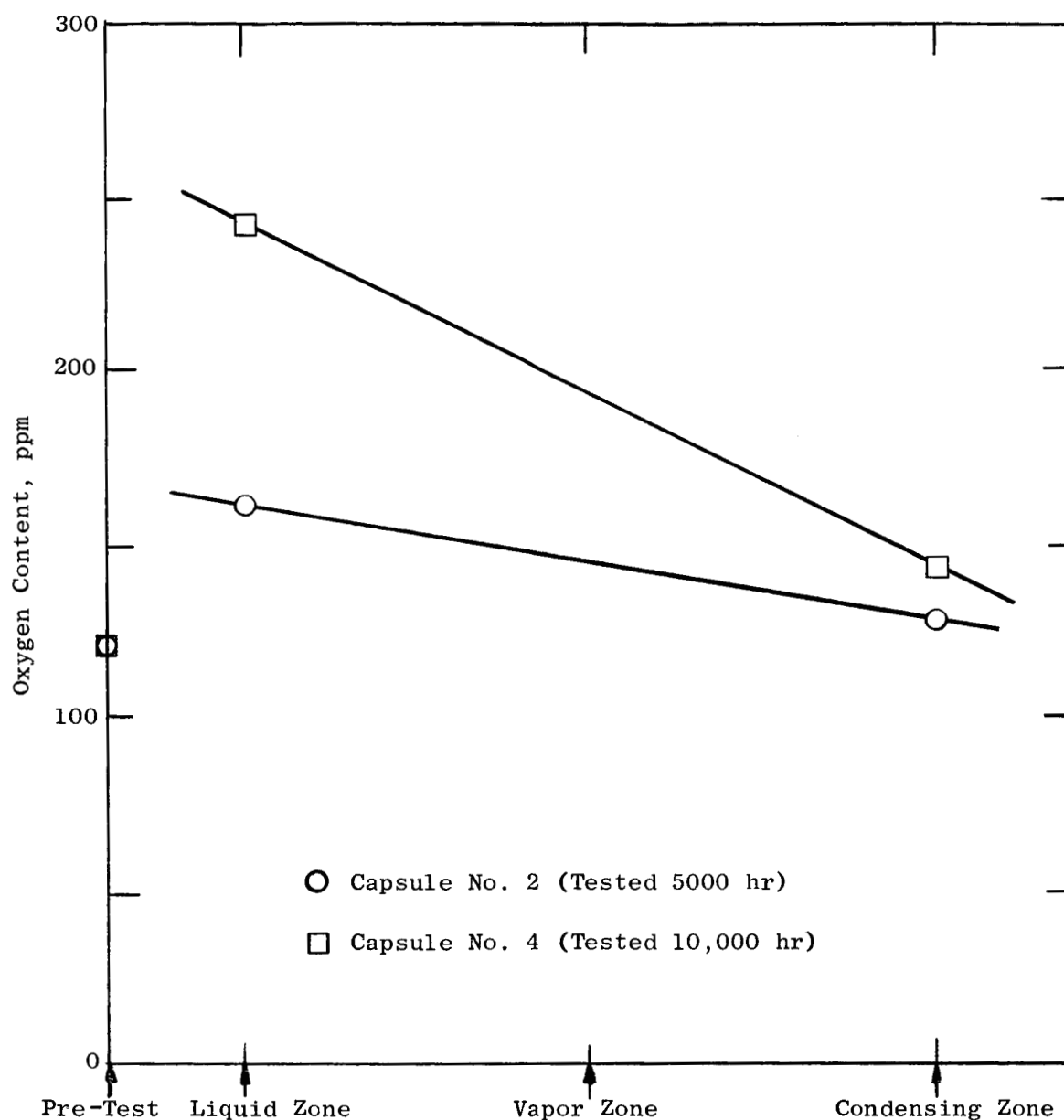


Figure 26. Oxygen Content of Inner 0.027-In. Layer of D-43 Alloy Capsule Wall as a Function of Specimen Location (Capsules Exposed to Potassium at 2000°F in a Vacuum of 10^{-9} Torr).

Metallographic Examination. The metallographic examination of the capsules was conducted on samples from the liquid zone, the liquid-vapor interface, and the condensing zone. Figure 23 cites the location of the samples.

No significant attack was observed in either the base metal (Figure 27) or the welds of any of the capsules. Micrographs of capsule welds in the condensing zones in Figure 28 are typical of the weld structures observed in the other regions of the capsules. The large grain sizes in the welds of the Cb-1Zr and D-43 alloys are quite evident when compared to the smaller grain size of the weld in the AS-55 alloy.

A grain boundary precipitate, observed in the Cb-1Zr alloy after 10,000-hr testing, is probably ZrO_2 ; an electron microprobe analysis indicated that the particles were rich in zirconium (Figure 29). Re-examination of the metallographic specimens from Cb-1Zr alloy capsule No. 6 which was exposed for 5000 hr revealed a similar but, as might be expected, less extensive precipitate.

Relatively large grains observed, after testing, on the inside diameter of Cb-1Zr alloy capsule No. 7 in the condensing zone (Figure 30) are believed to result from accelerated grain growth following the previously discussed loss of oxygen and zirconium from the condensing zone. No grain boundary precipitate was observed near the inside diameter in the condensing zone of Cb-1Zr alloy capsule No. 7; this correlates with an anticipated shift in the $ZrO_2=Zr+2O$ equilibria as the result of the decreasing zirconium and oxygen content in the columbium. This type grain growth was not observed in Cb-1Zr alloy capsule No. 6 tested for 5000 hr.

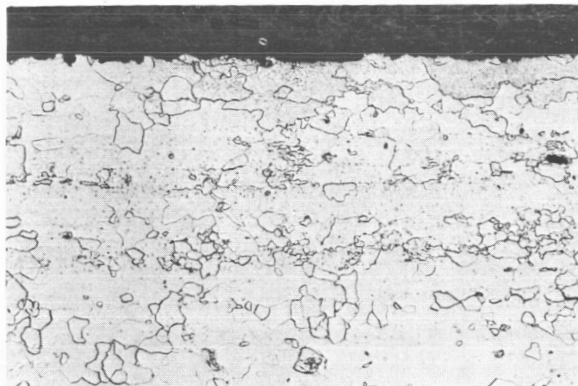
The same morphological change observed in AS-55 alloy capsules exposed for 5000 hr was observed in the AS-55 alloy capsules exposed for 10,000 hr (Figure 31). The Widmanstätten carbide structure seen in the matrix of the pre-test AS-55 alloy sheet material has gone into solution and a new precipitate has formed in the grain boundaries. Stain etching techniques and electron microprobe analysis tentatively identified the grain boundary precipitate as a columbium-zirconium carbide.

A grain boundary precipitate was observed in D-43 alloy capsule No. 4 after 10,000 hr of testing and, again, was tentatively identified as a columbium-zirconium carbide through stain etching techniques (Figure 32). A re-examination of D-43 alloy capsule No. 2, which was tested for 5000 hr, revealed a similar grain boundary precipitate.

Except the inside diameter of the condensing zone of Cb-1Zr alloy capsule No. 7, the other capsules evidenced no unusual grain growth as a result of the 10,000-hr exposure to refluxing potassium or high vacuum (10^{-9} torr).

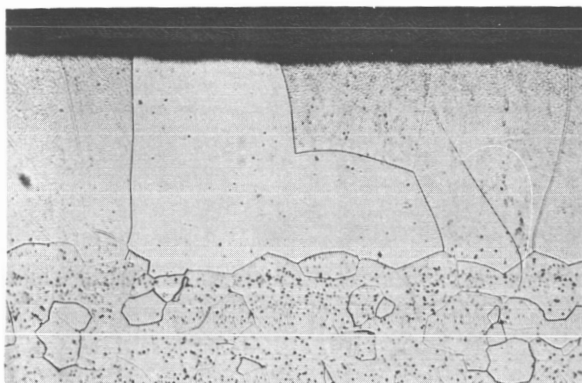
Cb-1Zr Alloy Capsule No. 7
High Oxygen Potassium

Transverse Section
of Annealed 0.080-
In. Thick Sheet
Before 10,000-Hr
Exposure



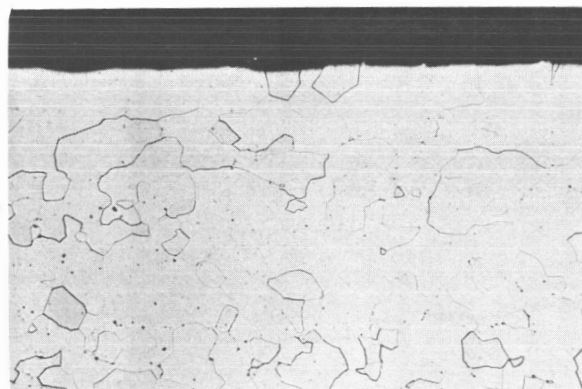
(A370111)

Transverse Section
of Condensing Zone
After 10,000-Hr
Exposure



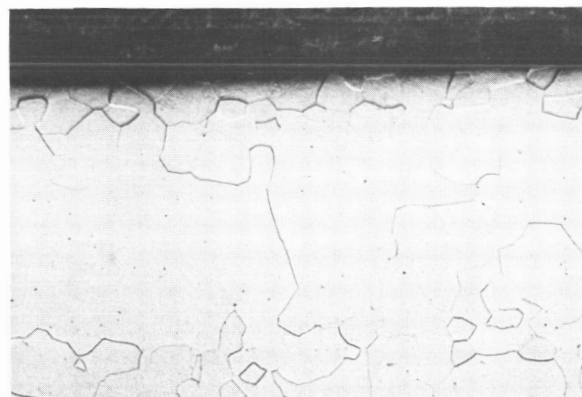
(A350111)

Longitudinal Section
of Liquid-Vapor Inter-
face After 10,000-Hr
Exposure



(A370311)

Transverse Section
of Liquid Zone After
10,000-Hr Exposure

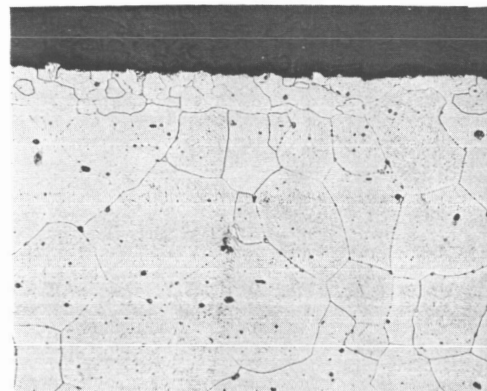


(A370411)

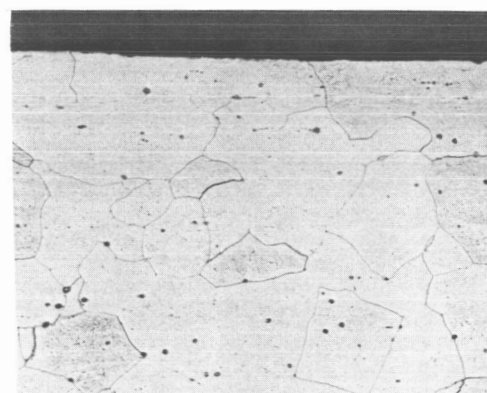
AS-55 Alloy Capsule No. 9
High Oxygen Potassium



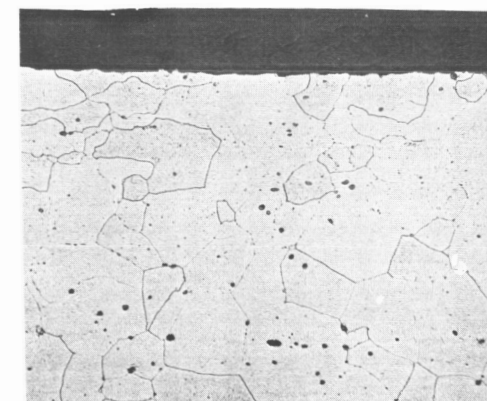
(K661)



(K100)



(K495)

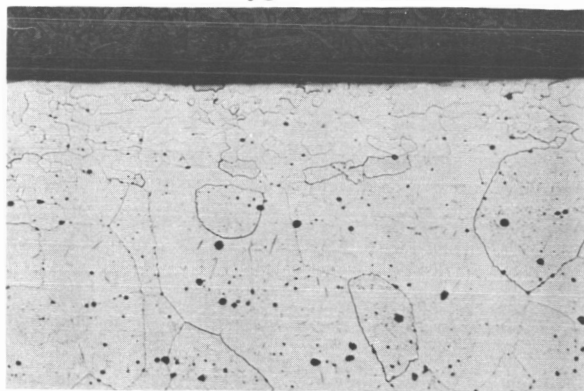


(K176)

Figure 27. Microstructures of AS-55, D-43, and Cb-1Zr Alloy to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

Etchant: 60% Glycerine, 20% HNO₃, 20% HF

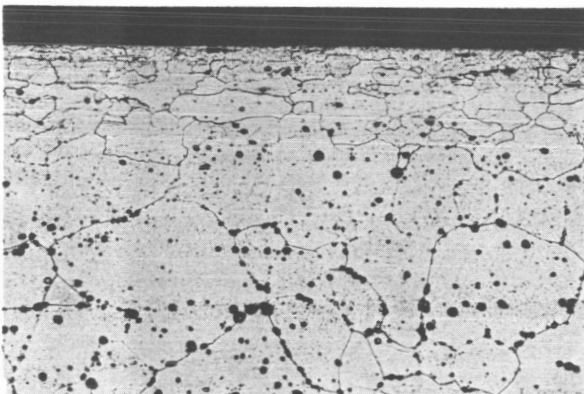
AS-55 Alloy Capsule No. 11
Low Oxygen Potassium



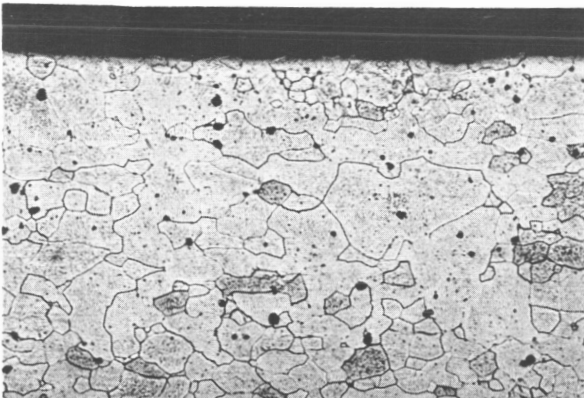
(A070511)



(A070611)

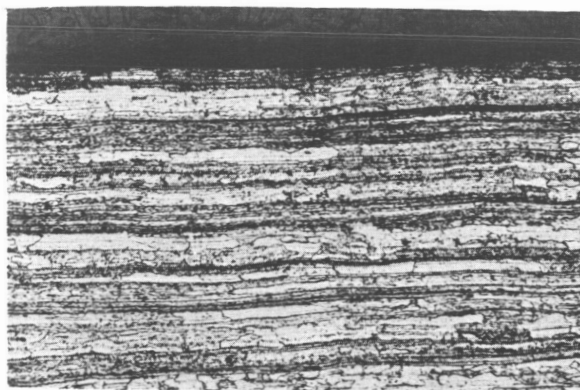


(A070711)

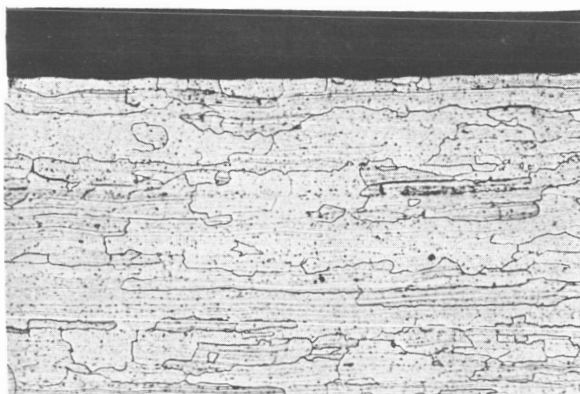


(A070811)

D-43 Alloy Capsule No. 4
Low Oxygen Potassium



(A390112)



(A390211)



(A390312)



(A390411)

0 1 2 3 4
Mils

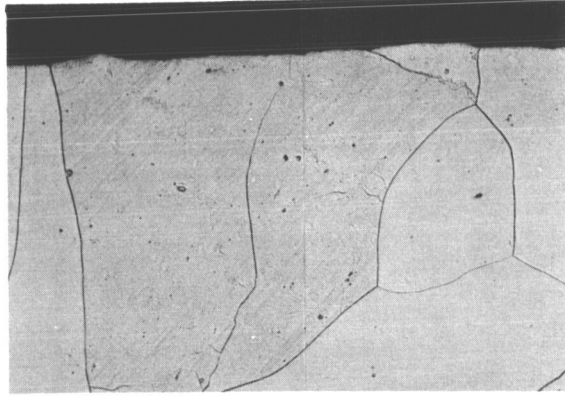
Reflux Corrosion Capsules Before and After 10,000-Hr Exposure

Orig. Mag.: 250X

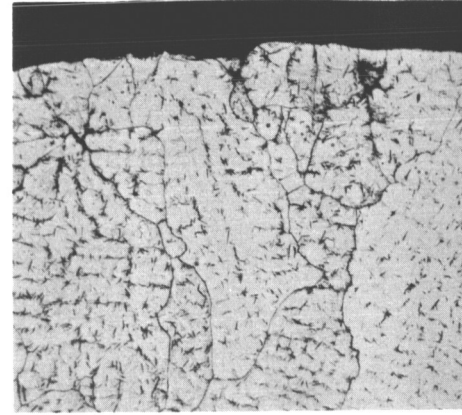
Cb-1Zr Alloy Capsule No. 7
High Oxygen Potassium

AS-55 Alloy Capsule No. 7
High Oxygen Potassium

Transverse Section
of Weld Before
10,000-Hr Exposure

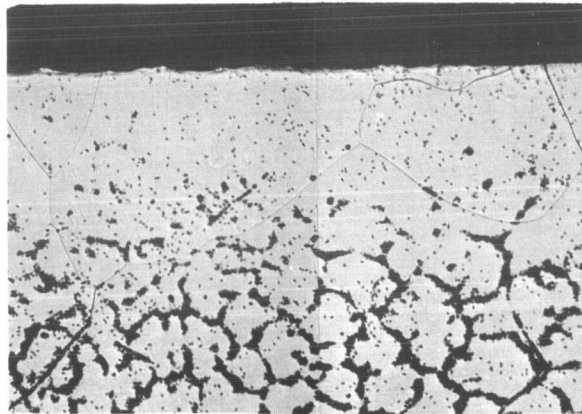


(K705)

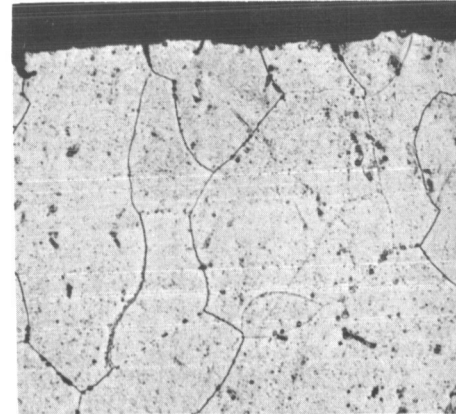


(K660)

Transverse Section
of Weld in Condensing
Zone After 10,000-Hr
Exposure

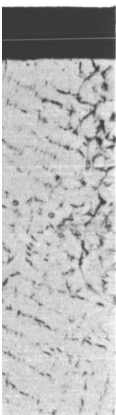


(A150111)

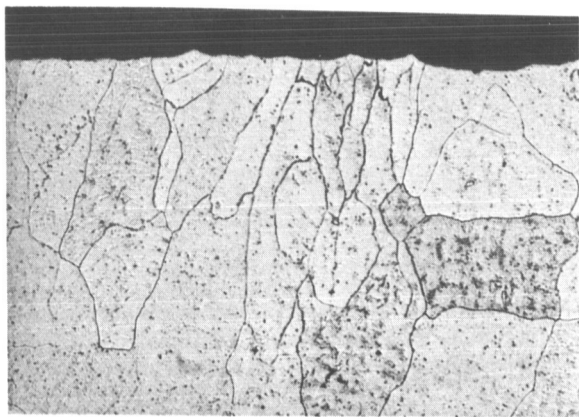


(A150211)

Figure 28. Microstructure of Welds in AS-55, D-43, and Cb-1Zr Alloy Capsules After Exposure to Potassium at 2000°F in a Vacuum of 10⁻⁶ Torr. Etchant: 60% Glycerine, 20% HNO₃, 20% HF



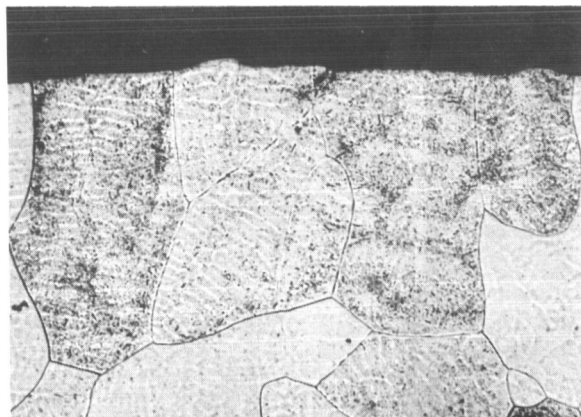
AS-55 Alloy Capsule No. 11
Low Oxygen Potassium



(A150311)

0 1 2 3 4
Mils

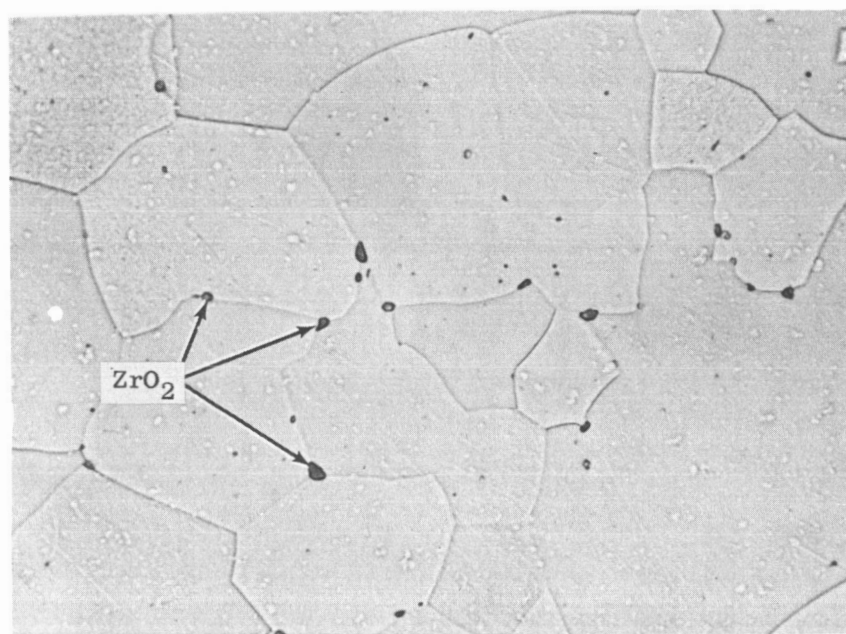
D-43 Alloy Capsule No. 4
Low Oxygen Potassium



(A390212)

Al Alloy Reflux Corrosion Capsules Before and After 10,000-Hr
 10^{-9} Torr.

Orig. Mag.: 250X



(A370313)

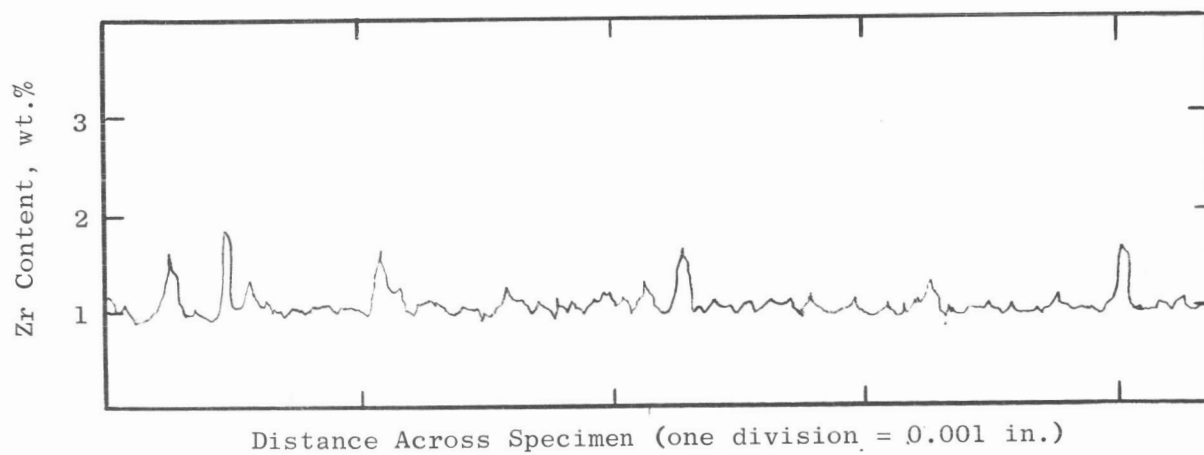
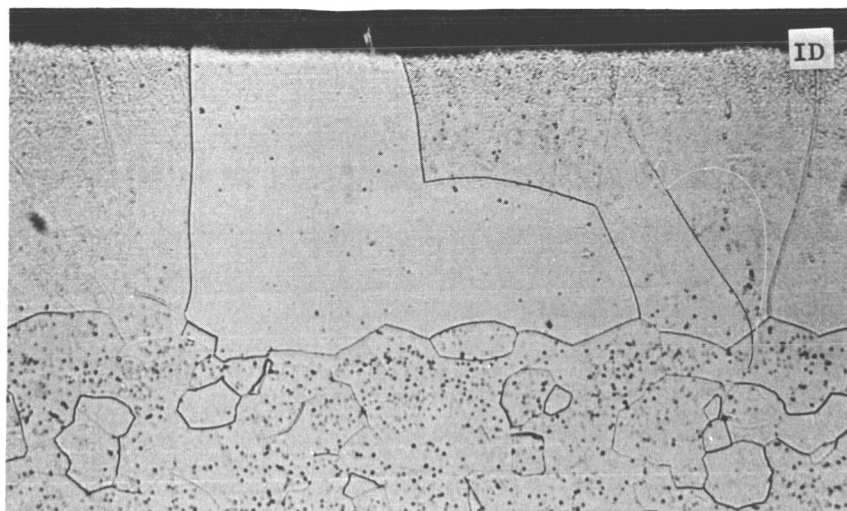


Figure 29. Photomicrograph and Electron Microprobe Analysis of Precipitate in Cb-1Zr Alloy Reflux Corrosion Capsule No. 7 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

Etchant: 60% Glycerine, 20% HNO_3 , 20% HF

Mag.: 1000X
N.A.: 0.85



(A350111)

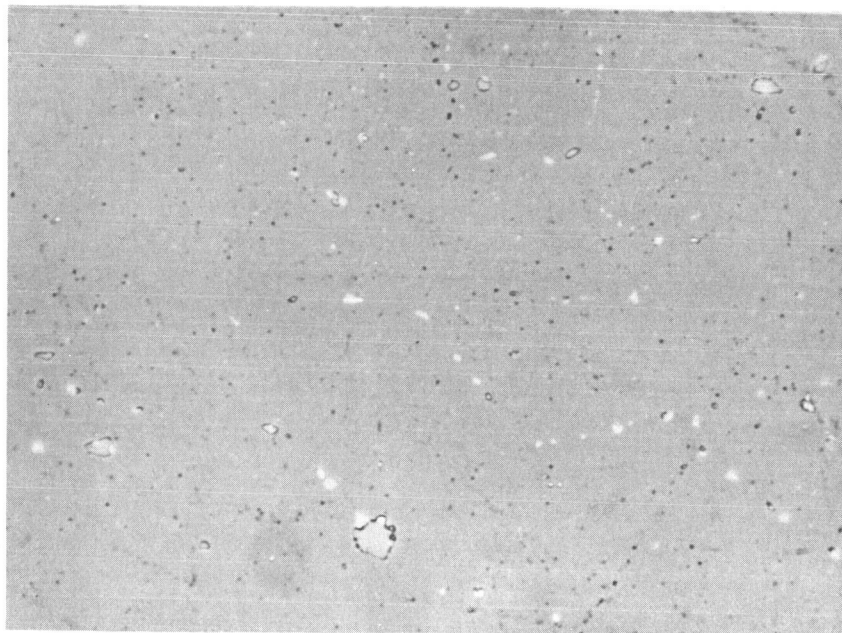


(A350112)

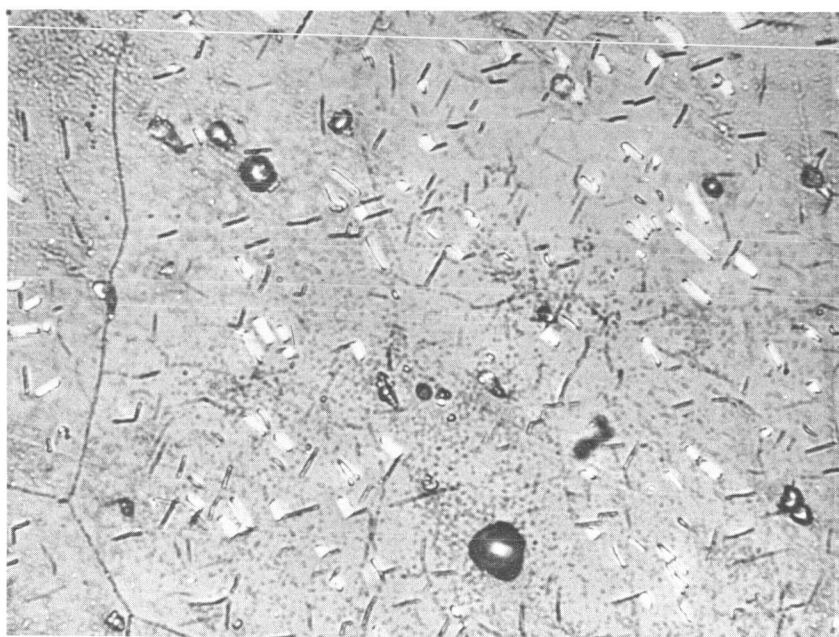
Figure 30. Microstructure in Condensing Zone of Cb-1Zr Alloy Reflux Corrosion Capsule No. 7 After 10,000-Hr Exposure to Potassium at 2000^oF in a Vacuum of 10⁻⁹ Torr.

Etchant: 60% Glycerine, 20% HNO₃, 20%HF

Mag. Top: 250X
Bottom: 50X



Post Test (A160111)



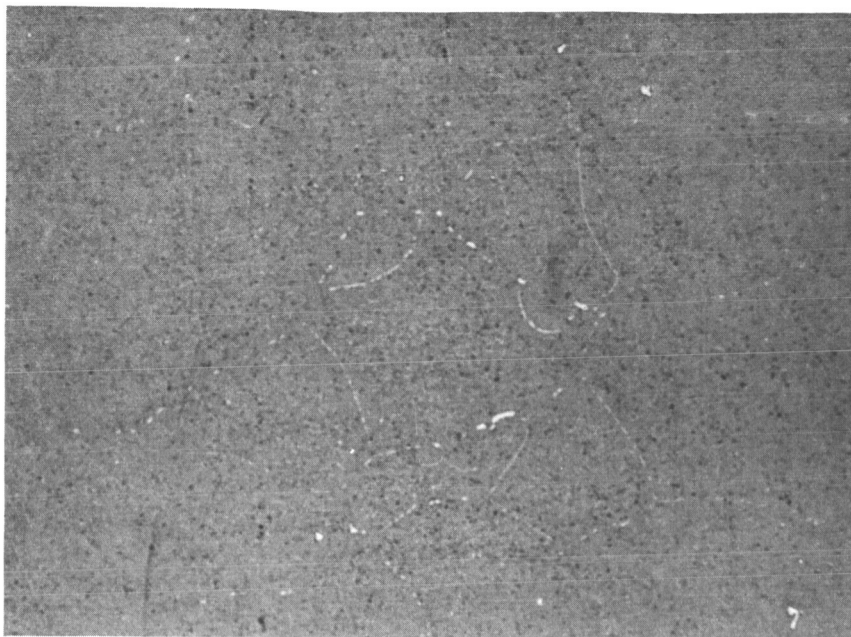
Pre-Test (A070111)

Figure 31. Microstructure of AS-55 Alloy Reflux Corrosion Capsule Before and After 10,000-Hr Exposure to Potassium at 2000 F in a Vacuum of 10^{-9} Torr.

Etchant: Stain Etched

Mag.: 1000X

N.A.: 0.85



Post Test (A390413)



Pre-Test (A390111)

Figure 32. Microstructure of D-43 Alloy Reflux Corrosion Capsule Before and After 10,000-Hr Exposure to Potassium at 2000^oF in a Vacuum of 10⁻⁹ Torr.

Etchant: Stain Etched

Mag.: 1000X

N.A.: 0.35

Overall, results of the 10,000-hr reflux capsule corrosion tests are similar to those of the 5000-hr tests.

Hardness Measurements. Transverse microhardness surveys were made across the capsule walls (Figures 33 through 36). A comparison of the hardness data obtained for the Cb-1Zr alloy and AS-55 alloy capsules tested for 10,000 hr showed no significant differences in hardness as compared with the results obtained from the 5000-hr tests. The loss in hardness in the Cb-1Zr alloy capsules is attributed to recovery/recrystallization phenomenon. Again, the AS-55 alloy capsules exhibited a significant decrease in hardness which would follow the carbide solution and reprecipitation previously discussed as well as from the loss in oxygen due to the exposure to potassium. The D-43 alloy capsule No. 4 tested for 10,000 hr showed a slightly larger decrease in hardness than that observed in the D-43 alloy capsule No. 2 tested for 5000 hr. This decrease in hardness in the D-43 alloy capsules is attributed to carbide precipitation in the grain boundaries and recovery/recrystallization during testing, since the material was not completely recrystallized before the initiation of the test.

Weight and Thickness Measurements. The weight and thickness changes (Table VIII) that ensued from subjecting the bend specimens to the potassium environment within the capsules are quite small and considered insignificant.

Bend Tests. All the bend specimens, which were exposed to potassium liquid and vapor, successfully withstood a 1 "T", or smaller, bend through a 105-degree bend angle at room temperature.

Stress-Rupture Tests. Two stress-rupture specimens were machined from the walls of AS-55 capsules Nos. 9 and 11, two from D-43 capsule No. 4, and two from Cb-1Zr capsule No. 7 in such a manner that the location of the gauge section of one specimen of each capsule is in the liquid region and the location of the gauge length of the second specimen is in the condensing region. The two specimens from AS-55 capsule No. 11 were machined from the inner 0.070-in. thick portion of the capsule wall; all other specimens, from the entire capsule wall. In an effort to eliminate the possible strengthening effect of surface contamination resulting from Al_2O_3 grit blasting of the outside diameter of the capsules, the outer 0.010 in. was removed from the specimen from AS-55 capsule No. 11.

All specimens were tested at 2000°F. The results of these tests and the tests previously reported⁷ on capsules exposed for 5000 hr are presented in Table IX; corresponding Larson-Miller parameter plots are shown in Figures 37, 38, and 39. An additional stress-rupture test (discussed in reference 2) of a specimen machined from the condensing zone of AS-55 capsule No. 12, which was exposed for

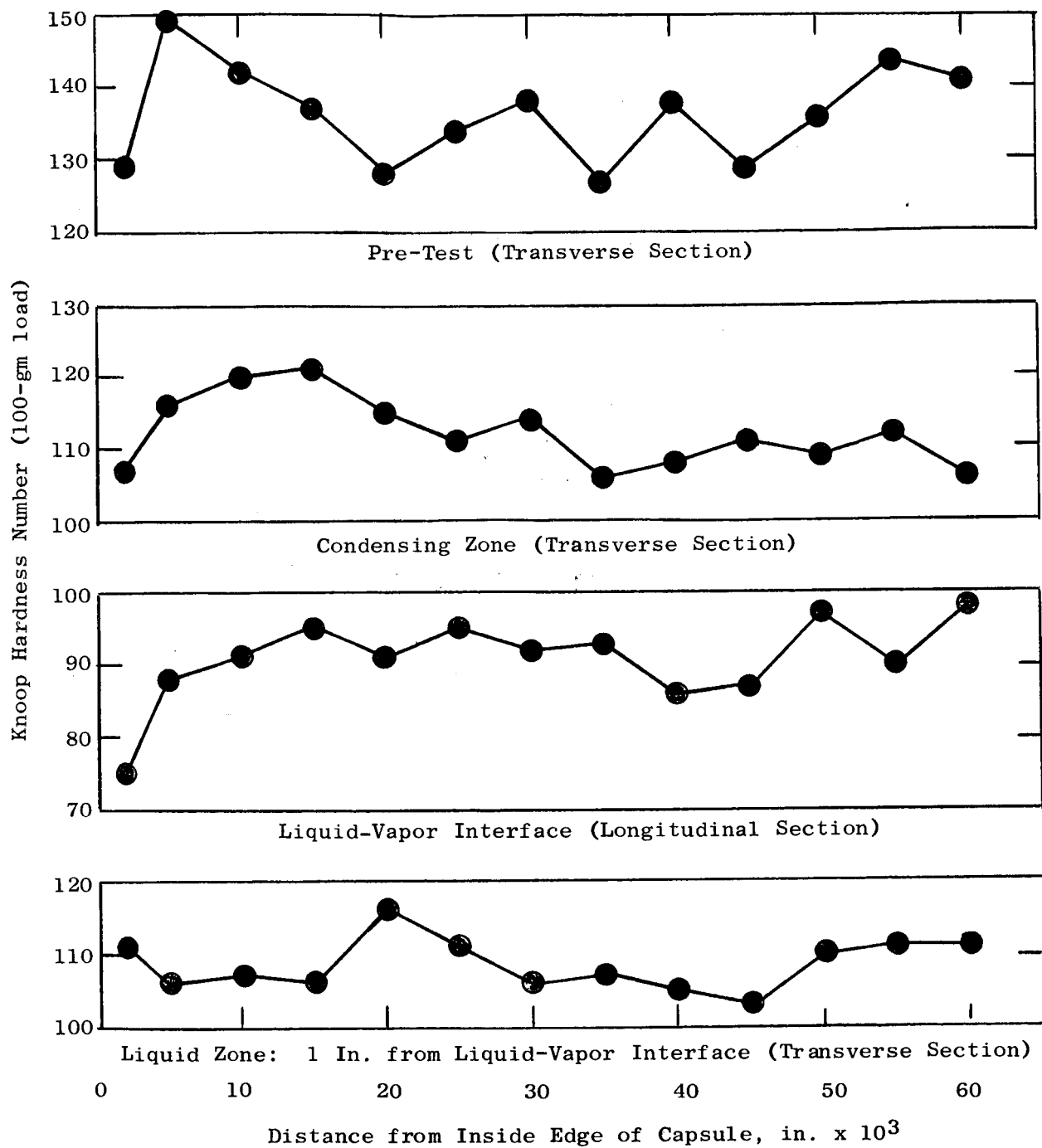


Figure 33. Microhardness Traverses of Cb-1Zr Alloy Reflux Corrosion Capsule No. 7 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10⁻⁹ Torr.

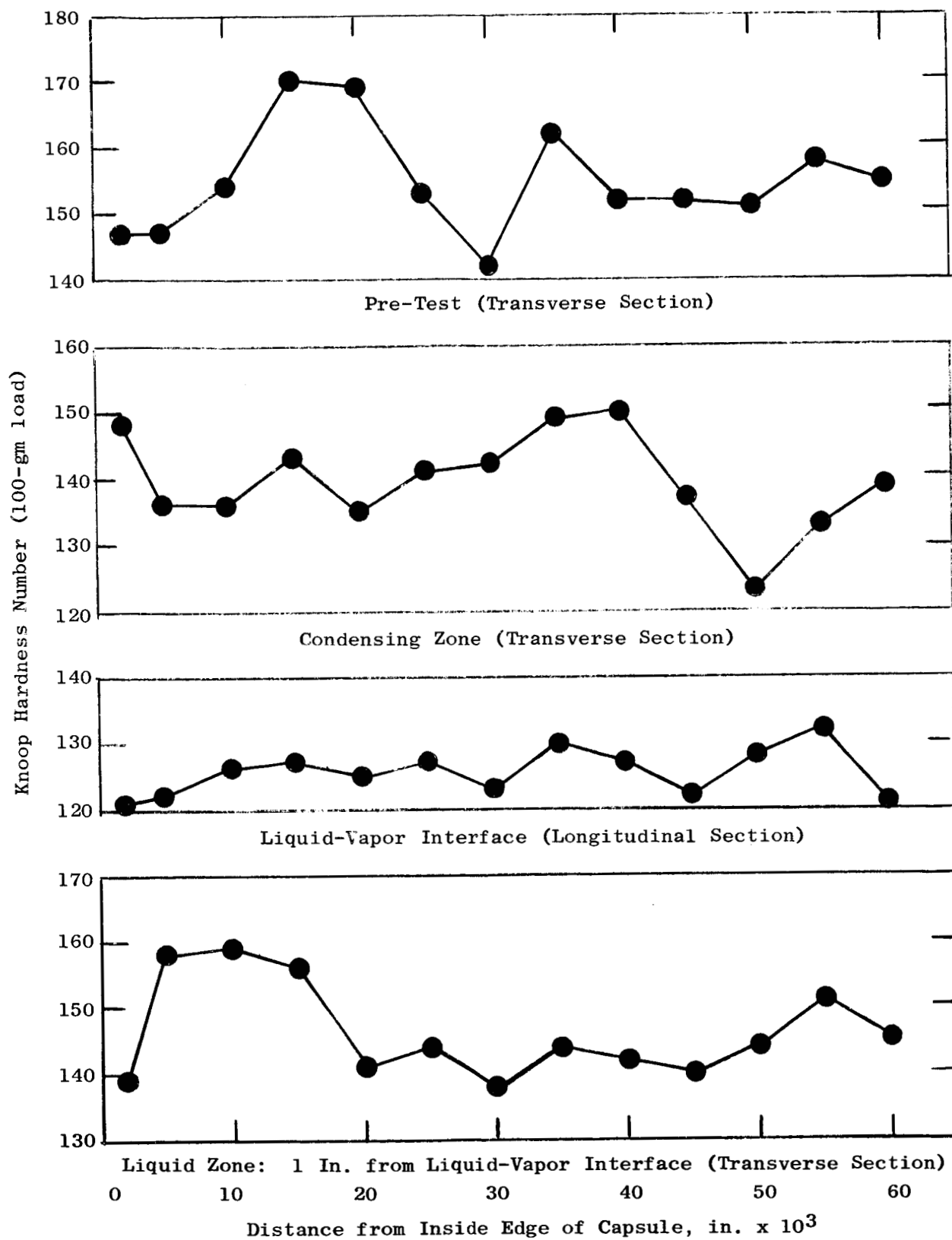


Figure 34. Microhardness Traverses of AS-55 Alloy Reflux Corrosion Capsule No. 9 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum at 10⁻⁹ Torr.

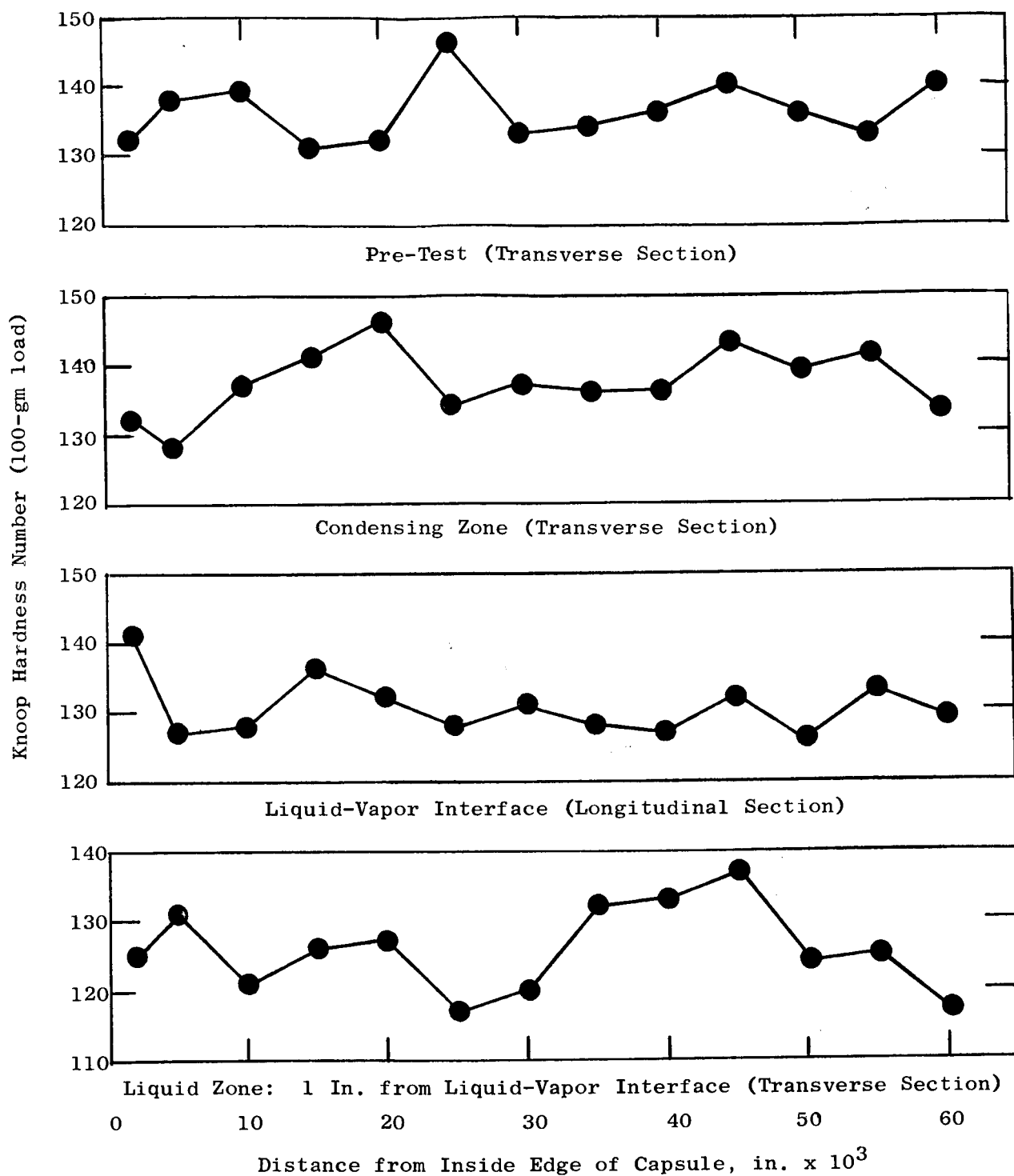


Figure 35. Microhardness Traverses of AS-55 Alloy Reflux Corrosion Capsule No. 11 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

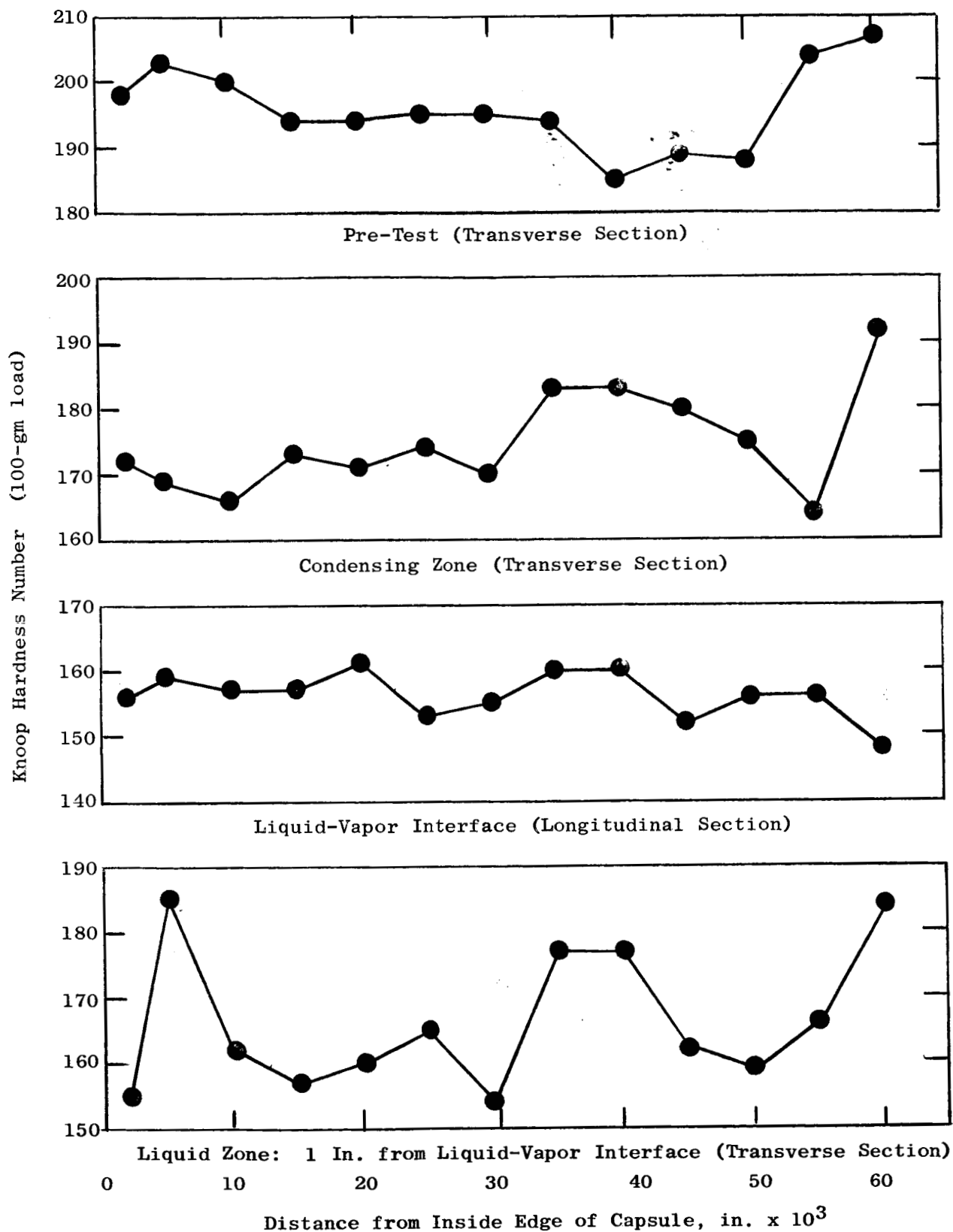


Figure 36. Microhardness Traverses of D-43 Alloy Reflux Corrosion Capsule No. 4 After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

Table VIII. Thickness and Weight Measurements of Bend Specimens Located in Reflux Corrosion Capsules and Exposed to Potassium for 10,000 Hr at 2000°F

Capsule	Thickness, in. ^a		Weight, gm		Wt. Change, mg/cm ²
	Before Exposure	After Exposure	Change	Before Exposure	After Exposure
No. 7 (Cb-12r)					
Liquid	0.0831	0.0829	-0.0002	b	b
Vapor	0.0842	0.0837	-0.0005	13.9481	13.9474
					-0.0007
					-0.0542
No. 9 (AS-55)					
Liquid	0.0508	0.0504	-0.0004	b	b
Vapor	0.0495	0.0493	-0.0002	8.7511	8.7510
					-0.0001
					-0.0077
No. 4 (D-43)					
Liquid	0.0584	0.0584	+0.0000	7.5614	7.5634
Vapor	0.0585	0.0585	+0.0000	7.9148	7.9249
					+0.0020
					+0.0101
					+0.1550
					+0.7827
No. 11 (AS-55)					
Liquid	0.0621	0.0615	-0.0006	7.4124	7.4130
Vapor	0.0614	0.0620	+0.0006		
					+0.0006
					+0.0465

^a Average of five measurements along specimen length.

^b No data; specimen welded to bottom of capsule.

Table IX. Stress-Rupture Data of Cb-1Zr, AS-55, and D-43 Alloys
Before and After 5000- and 10,000-Hr Exposures to Potassium at 2000°F
(Stress-Rupture Data Obtained at 2000°F)

Specimen	Test No.	Exposure Time, hr	Stress, psi	Press., torr	Rupture Life, hr	Elong./1 in., %	Larson-Miller Parameter, $T(15+\log t) \times 10^{-3}$
Cb-1Zr							
Capsule No. 7 ^a							
Liquid Zone	15	10,000	10,000	10 ⁻⁷	880.6 ^b	31.0	44.0
Condensing Zone	16	10,000	10,000	10 ⁻⁷	123.4 ^b	55.0	43.4 ^c
AS-55							
Before Exposure ^d	1	---	22,000	10 ⁻⁶	14.0	10.1	39.7
Before Exposure ^d	2	---	18,000	10 ⁻⁶	43.0	7.0	41.0
Before Exposure ^d	3	---	15,000	10 ⁻⁶	113.0	5.8	42.0
Capsule No. 8 ^e							
Liquid Zone	7	5,000	15,000	10 ⁻⁸	671.0 ^f	1.6	43.8 ^f
Condensing Zone	8	5,000	15,000	10 ⁻⁸	183.0	19.8	42.4
Capsule No. 12 ^g							
Condensing Zone	17	5,000	15,000	10 ⁻⁸	9.1	16.0	39.2
Capsule No. 9 ^h							
Liquid Zone	11	10,000	15,000	10 ⁻⁸	13.7 ^f	10.2	39.6 ^f
Condensing Zone	12	10,000	15,000	10 ⁻⁷	619.0 ^f	3.8	43.7 ^f
Capsule No. 11 ⁱ							
Liquid Zone	18	10,000	15,000	10 ⁻⁸	16.4	13.0	39.8
Condensing Zone	19	10,000	15,000	10 ⁻⁷	9.6	12.0	39.4
D-43							
Before Exposure ^j	4	---	25,000	10 ⁻⁶	51.0	14.8	41.1
Before Exposure ^j	5	---	22,000	10 ⁻⁶	89.0	20.4	41.6
Before Exposure ^j	6	---	20,000	10 ⁻⁶	118.0	26.7	42.0
Capsule No. 2 ^k							
Liquid Zone	9	5,000	20,000	10 ⁻⁸	140.0	28.8	42.1
Condensing Zone	10	5,000	20,000	10 ⁻⁸	382.0	30.5	43.2
Capsule No. 4 ^m							
Liquid Zone	13	10,000	20,000	10 ⁻⁷	18.1	31.0	39.8
Condensing Zone	14	10,000	20,000	10 ⁻⁸	11.6	29.0	39.4

^aCb-1Zr alloy (Heat No. 510); specimens machined from wall of reflux capsule No. 7; test conducted in getter-ion pumped system.

^bElement short caused 250°F overtemperature from 116 hr.

^cParameter calculated for 466 hr at 2000°F (since parameter for 7.4 hr at 2250°F is equivalent to 350 hr at 2000°F).

^dAS-55 alloy (NAS-5515); 0.060-in. thick sheet; heat-treated 1 hr/2800°F + 1 hr/2400°F; tests conducted in liquid nitrogen trapped, oil diffusion pumped system.

^eAS-55 alloy (NAS-5514); specimens machined from wall of reflux capsule No. 8; tests conducted in getter-ion pumped system.

^fSpecimen did not fail.

^gAS-55 alloy (NAS-5515); specimens machined from inner 0.070-in. thick portion of wall of reflux capsule No. 12; test conducted in getter-ion pumped system.

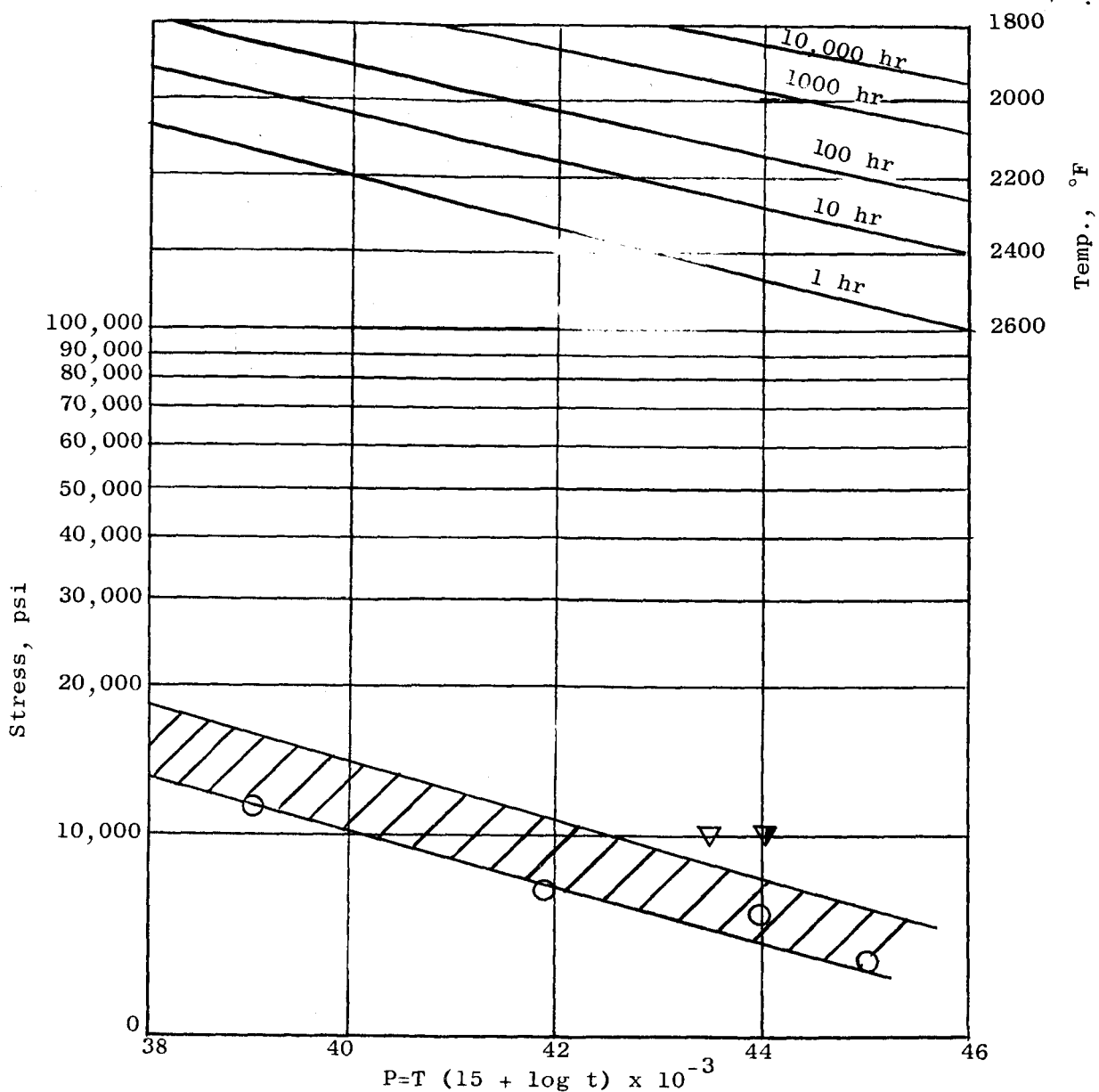
^hAS-55 alloy (NAS-555); specimens machined from wall of reflux capsule No. 9; test conducted in getter-ion pumped system.

ⁱAS-55 alloy (NAS-5515); specimens machined from inner 0.070-in. thick portion of wall of reflux capsule No. 11; tests conducted in getter-ion pumped system.

^jD-43 alloy (D-43-322); 0.055-in. thick sheet; heat-treated 1 hr/2200°F + 1 hr/2400°F; tests conducted in liquid nitrogen trapped, oil diffusion pumped system.

^kD-43 alloy (D-43-322); specimens machined from wall of reflux capsule No. 2; tests conducted in getter-ion pumped system.

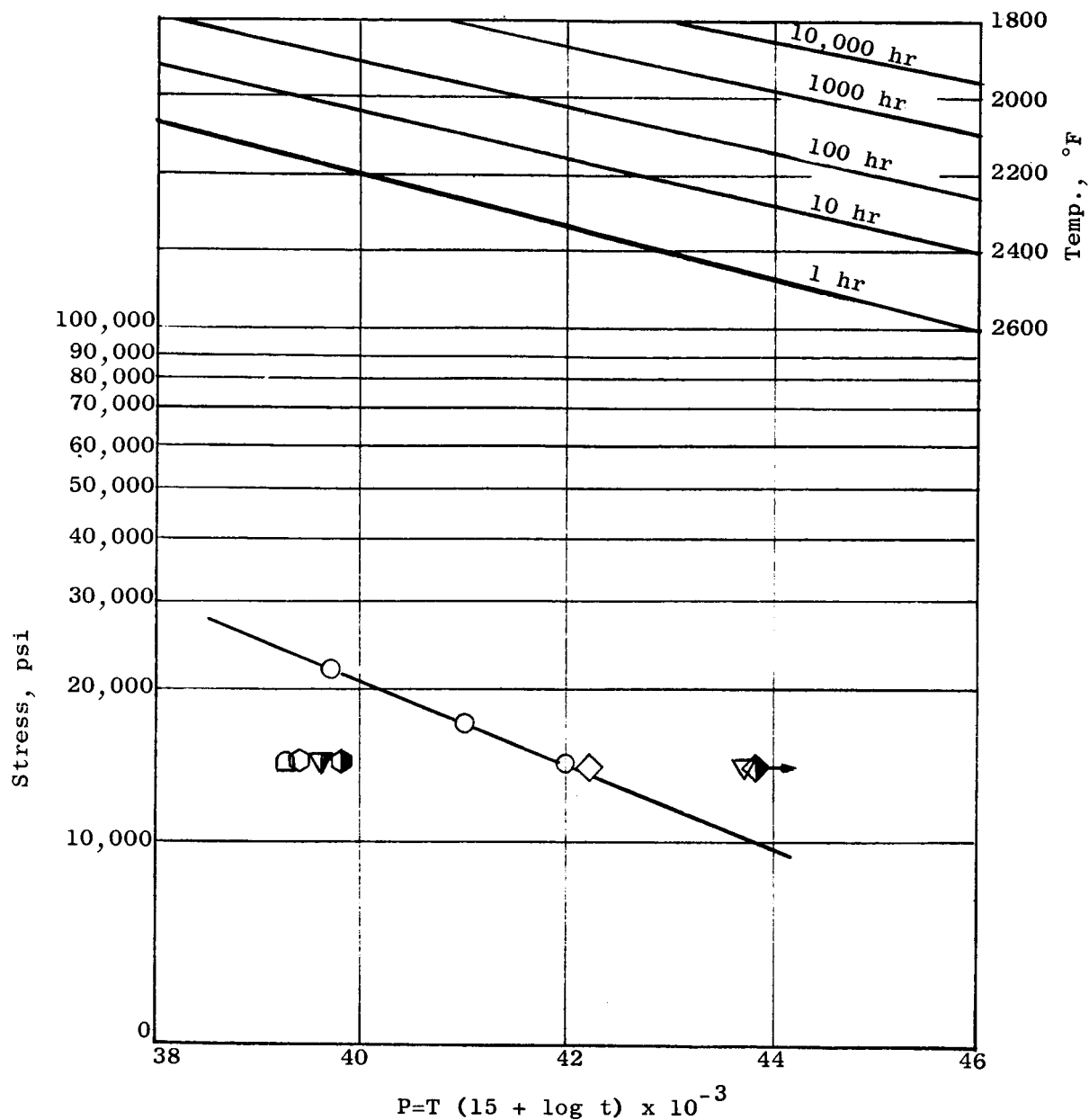
^mD-43 alloy (D-43-322); specimens machined from wall of reflux capsule No. 4; test conducted in getter-ion pumped system.



Typical stress-rupture properties of Cb-1Zr alloy (Cf. "Recent General Electric Co. Developments in Columbium-Base Alloys," FPL-501-2, Feb. 1962).

- Rupture properties of 0.060-in. thick Cb-1Zr alloy sheet (Cf. H.E. McCoy, "Creep Properties of the Nb-1% Zr Alloy," J. Less Common Metals, 8, pp. 20-35, 1965).
- ▽ Cb-1Zr alloy (519); specimen machined from wall of condensing zone of reflux capsule no. 7; rupture test performed in a vacuum of 10^{-7} torr.
- ▼ Cb-1Zr alloy (519); specimen machined from wall of liquid zone of reflux capsule no. 7; rupture test performed in a vacuum of 10^{-7} torr.

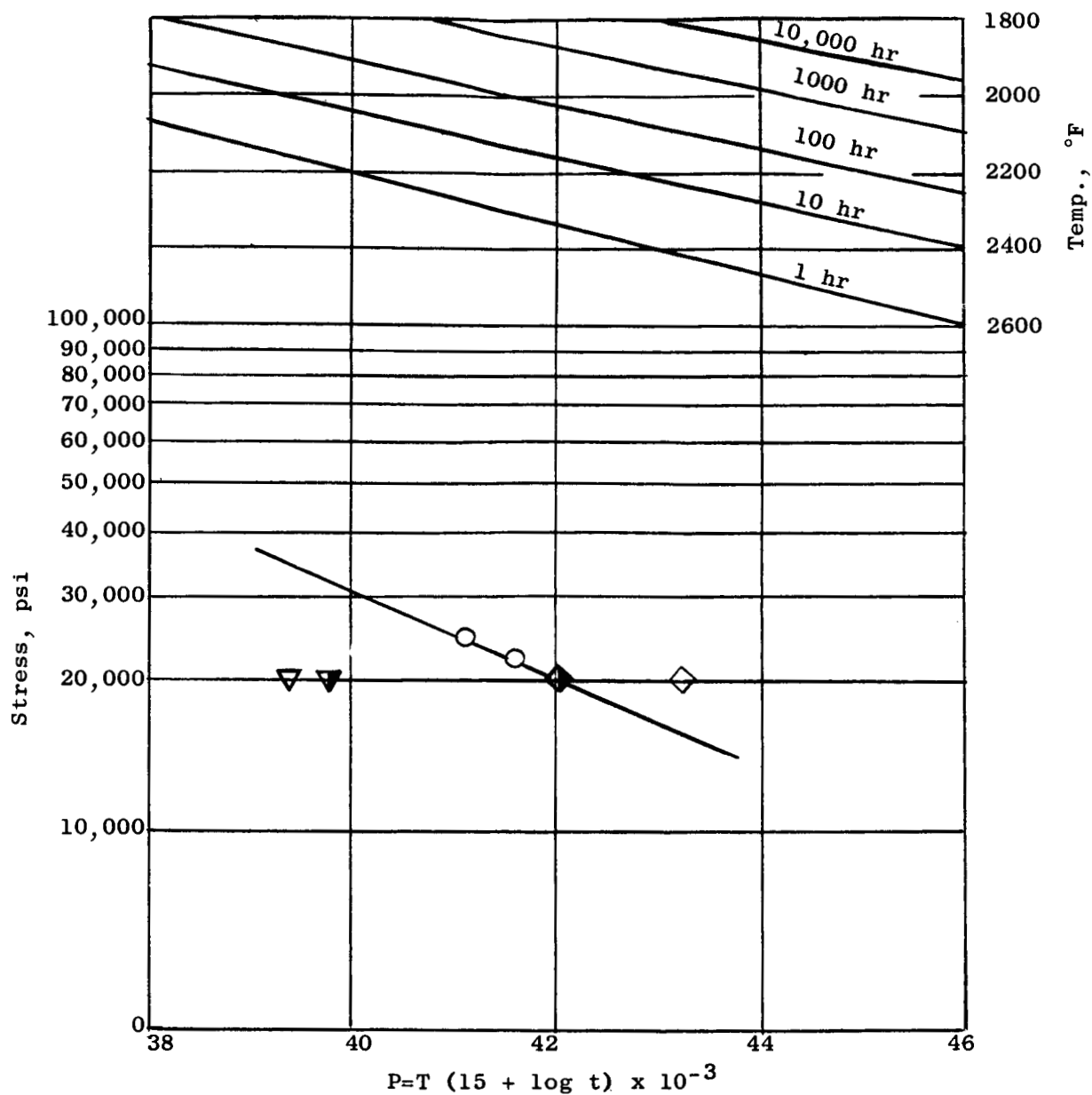
Figure 37. Stress-Rupture Properties of Cb-1Zr Alloy Before and After 10,000-Hr Exposure to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.



Corresponding data for test numbers cited below are presented in Table IX:

- | | |
|---------------------|---------------|
| ○ Test Nos. 1, 2, 3 | ▼ Test No. 11 |
| ◆ Test No. 7 | ▽ Test No. 12 |
| ◇ Test No. 8 | ◼ Test No. 18 |
| □ Test No. 17 | ◻ Test No. 19 |

Figure 38. Stress-Rupture Properties of AS-55 Alloy Before and After 5000- and 10,000-Hr Exposures to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.



Corresponding data for test numbers cited below are presented in Table IX:

- Test Nos. 4, 5, 6
- ◆ Test No. 9
- ◇ Test No. 10
- ▼ Test No. 13
- ▽ Test No. 14

Figure 39. Stress-Rupture Properties of D-43 Alloy Before and After 5000-and 10,000-Hr Exposures to Potassium at 2000°F in a Vacuum of 10^{-9} Torr.

5000 hr, also was performed and the results are included in Table IX and Figure 38. This specimen from AS-55 capsule No. 12 was machined from the inner 0.070-in. thick portion of the capsule in a similar manner and for the same reason (i.e., to eliminate the possible strengthening effect of grit blasting as discussed earlier) as the specimens from AS-55 capsule No. 11.

Data was obtained from specimens Nos. 1 through 6 (Table IX), which had not been exposed to potassium, in a liquid nitrogen trapped, cold-wall oil diffusion pumped, stress-rupture facility that can achieve a pressure in the 10^{-6} torr range at the test temperature. Stress-rupture data was obtained from specimens Nos. 7 through 16 and 17 through 19, which had been exposed to potassium, in getter-ion pumped high vacuum stress-rupture facilities with pumping speeds of 400 ℓ /sec and 100 ℓ /sec, respectively (Figure 40). These two facilities can achieve pressures in the 10^{-9} torr range, when cold, and in the 10^{-8} torr range, when testing refractory metals at temperatures on the order of 2000°F. To determine the extent of environmental contamination, chemical analyses for the interstitial elements were obtained on a number of specimens after stress-rupture testing. The data are presented in Table X.

With one exception, all the specimens machined from the walls of the AS-55 alloy and D-43 alloy reflux corrosion capsules after the 10,000-hr exposure to potassium exhibited significantly shorter rupture lives at 2000°F than the specimens machined from the untested sheet material used to fabricate the capsules. The long rupture life of the specimen machined from the condensing zone of capsule No. 9 (AS-55 alloy), probably, is the result of oxygen contamination from the Al_2O_3 grit blasting of the capsule outside diameter, as indicated by the high oxygen content in the outside portion of the capsule in the condensing zone. Both stress-rupture specimens from capsule No. 11 (AS-55 alloy), from which the outer, grit-blasted surface had been removed before testing, displayed a shorter rupture life than that of the starting material which can be attributed to carbide coalescence and the leaching of oxygen from the capsule inner wall.

The specimen (machined from the condensing zone of AS-55 alloy capsule No. 12, exposed for 5000 hr) subjected to the additional stress-rupture test also showed a shorter rupture life than that of the starting material, which correlates with the data obtained from AS-55 capsule No. 11, exposed for 10,000 hr. In the tests cited, the outer grit blasted surfaces were removed from the specimen prior to rupture testing. The results of these tests contrast with earlier data obtained on capsule No. 8 (AS-55 alloy exposed for 5000 hr), which showed a longer rupture life than the starting material. In this latter case, the specimens were machined from the entire cross section of the capsule wall, and the weakening effect of the observed carbide coalescence is masked by the increase in oxygen caused by

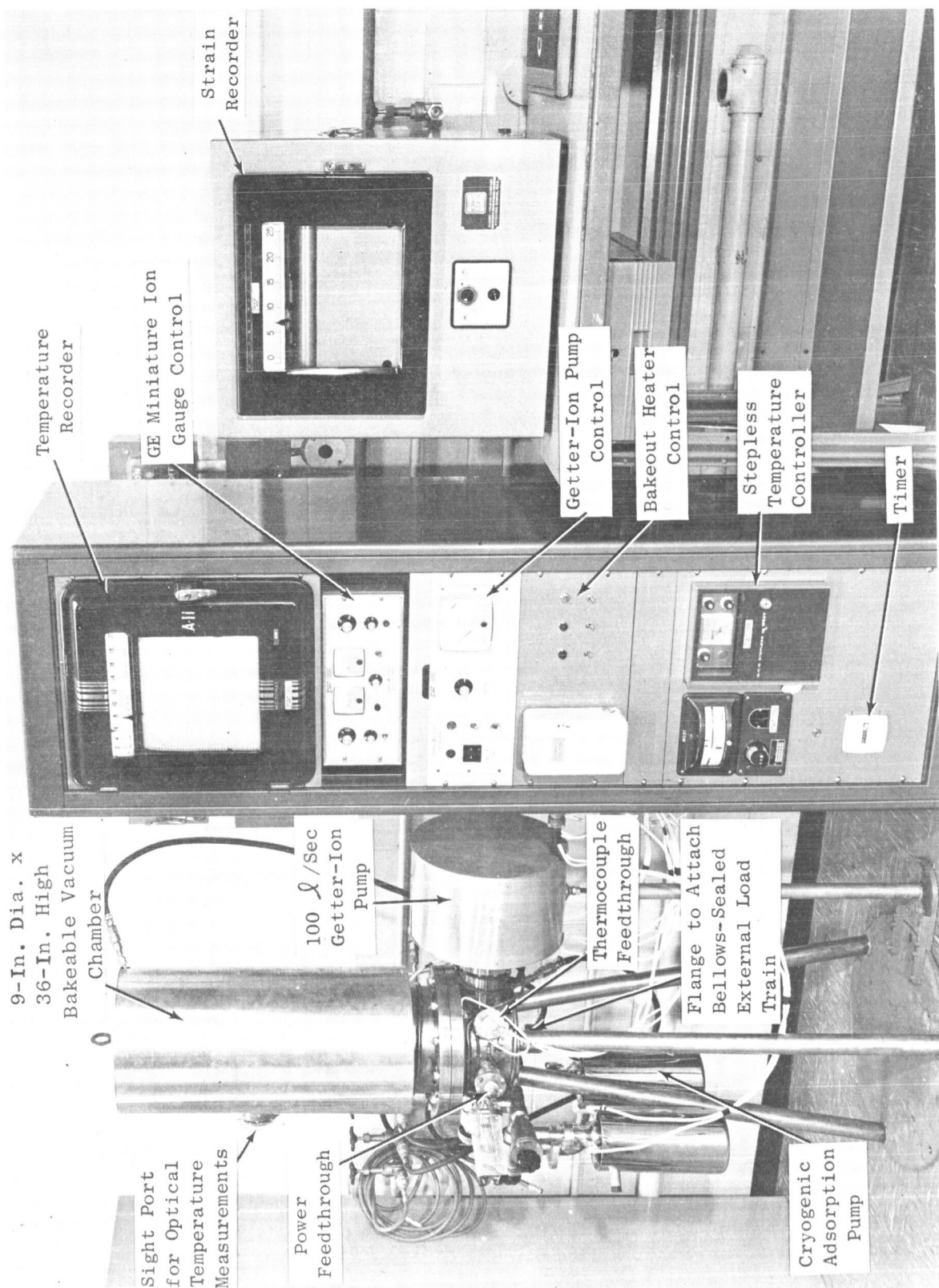


Figure 40. High Vacuum Stress-Rupture Facility, Equipped with 100 l/Sec Getter-Ion Pump, Capable of Achieving a Pressure of 10^{-9} Torr.

Table X. Chemical Analyses (Wt. %) of AS-55 and D-43 Alloys Before and After Stress - Rupture Testing at 2000°F in Vacuum

Specimen	Test No.	Rupture Life, hr	Press., torr	Chemical Analyses, wt. %									
				Before Test			After Test			O	C	H	C
				O	N	H	O	N	H				
AS-55 ^a	3	113	10 ⁻⁶	0.0356	0.0192	0.0002	0.106	0.0324	0.0213	0.0001	0.0001	0.065	
D-43 ^b	6	118	10 ⁻⁶	0.0110	0.0026	0.0004	0.101	0.0076	0.0023	0.0001	0.0001	0.084	
AS-55 ^c													
Liquid Zone	7	671 ^d	10 ⁻⁸	0.0498	0.0204	0.0008	0.060	0.0646	0.0237	0.0003	---	---	
D-43 ^e													
Condensing Zone	10	382	10 ⁻⁸	0.0138	0.0027	0.0004	0.108	0.0221	0.0050	0.0001	---	---	
AS-55 ^f													
Condensing Zone	12	619 ^d	10 ⁻⁷	0.0453	0.0185	0.0004	0.070	0.0365	0.0142	0.0002	---	---	
D-43 ^g													
Condensing Zone	14	11.6	10 ⁻⁸	0.0143	0.0032	0.0002	0.091	0.0264	0.0026	0.0001	---	---	

^aAS-55 alloy (NAS-5515); 0.060-in. thick sheet; heat-treated 1 hr/2800°F + 1 hr/2400°F; test conducted in liquid nitrogen trapped, oil diffusion pumped system.

^bD-43 alloy (D-43-322); 0.055-in. thick sheet; heat-treated 1 hr/2200°F + 1 hr/2400°F; test conducted in liquid nitrogen trapped, oil diffusion pumped system.

^cAS-55 alloy (NAS-5514); specimen machined from wall of reflux capsule No. 8; test conducted in getter-ion pumped system.

^dSpecimen did not fail.

^eD-43 alloy (D-43-322); specimen machined from wall of reflux capsule No. 2; test conducted in getter-ion pumped system.

^fAS-55 alloy (NAS-555); specimen machined from wall of reflux capsule No. 9; test conducted in getter-ion pumped system.

^gD-43 alloy (D-43-322); specimen machined from wall of reflux capsule No. 4; test conducted in getter-ion pumped system.

grit blasting of the outer capsule surface and possible contamination during rupture testing.

The rupture data obtained from specimens machined from the wall of capsule No. 4 (D-43 alloy) show that the rupture life of both the specimen from the liquid zone and the specimen from the condensing zone is shorter than the rupture life of the starting material before exposure to potassium. Also, the rupture life of the specimen from the liquid zone is slightly longer than that of the specimen from the condensing zone. These data would appear to substantiate the higher oxygen content in the liquid zone as compared to that in the condensing zone, a result of the gettering action during the exposure to potassium. A comparison between the rupture data obtained from specimens machined from capsule No. 4 (D-43 alloy), and the rupture data obtained earlier from No. 2 (D-43 alloy exposed for 5000 hr) showed no change in rupture life for the specimen from the liquid zone and a longer rupture life for the specimen from the condensing zone. However, coalescence of carbides was observed in all regions in both capsules Nos. 2 and 4.

The longer rupture lives of the specimens machined from the liquid and condensing zones of capsule No. 8 (Cb-1Zr alloy) are attributed to the increased oxygen concentration in the capsule wall caused by the gettering action in the liquid zone during the exposure to potassium and grit blasting of the outer capsule wall before the test exposure.

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APPENDIX

A. SUMMARY OF PROCESSING DETAILS OF AS-55, D-43, AND Cb-1Zr
ALLOY SHEET USED IN THE FABRICATION OF REFLUX CORROSION CAPSULES

Processing of AS-55 Alloy Heat NAS-555

1. Single consumable dc arc melt.
2. Cropped ingot dimensions: 2.9 in. in diameter x 4.45 in. long; 10.75 lb.
3. Ingot split lengthwise and one-half machined into forging billet.
4. Forging billet dimensions: 1.22 in. thick x 1.75 in. wide x 4.30 in. long; 2.87 lb.
5. Titanium clad forging billet dimensions: 1.333 in. thick x 3.93 in. wide x 6.43 in. long.
6. Clad billet soaked 45 min at 2200°F in argon.
7. Five blows on 2,500-lb forge hammer to 0.800-in. thick over-all 39% reduction.
8. Clad forging re-soaked 15 min at 2200°F in argon.
9. Clad forging rolled in the direction of the long axis of the ingot:

Pass	Thickness, in.	Red./Pass, %	Red. (Total), %
1	0.613	24	24
2	0.465	24	42
3	0.322	30	60

Soaked 15 min at 2200°F between passes.
Plate turned end for end each pass.

10. Stripped, pickled, and trimmed plate dimensions: 0.264 in. thick x 3.10 in. wide x 10.70 in. long; 2.7 lb.
11. One-third of plate sectioned and discarded (low stirring region of original ingot).
12. Plate cold rolled 65% to 0.080-in. thick sheet by successive 10-mil reductions.
13. Sheet stress-relieved one hr at 2300°F.

Processing of AS-55 Alloy Heat NAS-5514

1. Single consumable dc arc melt.
2. Cropped ingot dimensions: 3.05 in. in diameter x 4.37 in. long; 11.31 lb.
3. Ingot split lengthwise and one half-machined into forging billet.
4. Forging billet dimensions: 1.00 in. thick x 1.77 in. wide x 3.93 in. long; 2.25 lb.
5. Titanium clad billet dimensions: 1.124 in. thick x 4.37 in. wide x 6.50 in. long.
6. Clad billet soaked 45 min at 2200°F in argon.
7. Six blows on 2,500-lb forge hammer to 0.550-in. thick over-all 51% reduction.
8. Clad forging re-soaked 15 min at 2200°F in argon.
9. Clad forging rolled in direction of the long axis of the ingot on first pass, normal to the long axis on second pass, and returned to the original direction on final pass.

Pass	Thickness, in.	Red./Pass, %	Red. (Total), %
1	0.443	19	19
2	0.353	20	35
3	0.280	20	49

Soaked 15 min at 2200°F between passes.
Plate turned end for end each pass.

10. Stripped, pickled, and trimmed plate dimensions: 0.216 in. thick x 3.87 in. wide x 6.5 in. long; 1.68 lb.
11. Plate cold rolled 62% to 0.080-in. thick in successive 10-mil reduction.
12. Sheet stress-relieved one hr at 2300°F.

Processing of AS-55 Alloy Heat NAS-5515

1. Single consumable dc arc melt.
2. Cropped ingot dimensions: 3.65 in. in diameter x 10.3 in. long; 36.9 lb.
3. Ingot sectioned to remove defective area.
4. Billet sealed in molybdenum can.

5. Clad billet extruded 5.6:1 at 2600°F to 0.70-in. thick x 2.68 in. wide sheet bar.
6. Clad extrusion soaked 45 min at 2200°F in argon.
7. Clad extrusion rolled in direction perpendicular to extrusion direction:

Pass	Thickness, in.	Red./Pass, %	Red. (Total), %
1	0.632	10	10
2	0.546	13	22
3	0.454	16	35
4	0.360	20	48

Soaked 15 min at 2200°F between passes.
Plate turned end for end each pass.

8. Clad plate stripped, pickled, spot-ground and trimmed.
9. Plate cold rolled 75% to 0.082-in. thick sheet by successive 0.010-in. reductions.
10. Sheet stress-relieved one hr at 2200°F.

Processing of D-43 Alloy Heat D-43-322

1. Double consumable dc arc melt.
2. Ingot dimensions: 8 in. in diameter.
3. Billet extruded 4:1 at 2000°F to 2 in. thick x 6 in. wide sheet bar.
4. Extrusion warm rolled at 2000°F; temperature reduced gradually until sheet was 0.250-in. thick.
5. Plate annealed one hr at 2200°F.
6. Plate cold rolled to 0.130-in. thick sheet.
7. Sheet annealed one hr at 2200°F.
8. Sheet cold rolled to 0.080-in. thick sheet.
9. Sheet stress-relieved one hr at 2200°F.

Processing of Cb-1Zr Alloy Heat 519

1. Single electron beam melt.
2. Ingot dimensions: 5 in. in diameter x 20 in. long; 120 lb.
3. Ingot warm forged at 400°F to plate: 1.125 in. thick x 12 in. wide x 20 in. long.

4. Plate cold rolled to 0.100-in. thick sheet.
5. Sheet annealed one hr at 2200°F.

B. DATA SHEETS FOR THE CHEMICAL ANALYSES
OF POTASSIUM BY THE ZIRCONIUM-GETTER TECHNIQUE

Table I. Potassium Analyses by Zirconium-Gettering Technique
(Potassium sampled during filling of Cb-1Zr alloy capsules
Nos. 6 and 7 and AS-55 alloy capsules Nos. 8 and 9)

Auxiliary Capsule No.	Zr Sample Designation	Zr Analyses,				K Impurities (Calcd.),	
		ppm				ppm	
		O	N	H	C	O	C
2	a	1740	17	25	---	712	---
	b	1430	16	7	---	502	---
	c	2320	20	31	---	1104	---
	<u>d</u>	<u>1760</u>	<u>19</u>	<u>54</u>	<u>300</u>	<u>725</u>	<u>133</u>
	Av.	1813	18	29	300	760	133

Notes:

1. Auxiliary capsule exposed in a vacuum at 1400°F for 100 hr.
2. Cb-1Zr alloy reaction capsule one in. in diameter x 6 in. long x 0.080-in. thick wall.
3. Initial analyses of zirconium (ppm):

Sample	O	N	H	C
a	638	19	18	---
b	840	14	25	70
<u>c</u>	<u>587</u>	<u>31</u>	<u>40</u>	<u>135</u>
Av.	688	21	28	103
4. Auxiliary capsule No. 2 contained 7.14 gm potassium, 4.828 gm zirconium (20 in.² surface area).
5. Analysis of oxygen, nitrogen, and hydrogen in zirconium by vacuum fusion techniques.
6. Analysis of carbon in zirconium by conductometric techniques.
7. Potassium from Shipping Container A-4 slagged, filtered, distilled, and hot trapped.

Table II. Chemical Analyses of As-Received
Potassium from Shipping Container No. 137

Auxiliary Capsule No.	Zr Analyses,			K Impurities (Calcd.),		Remarks
	ppm			ppm		
	O	N	H	O	N	
3	1326	13	31.5	426	9	Potassium fill tube was not valved; capped in air.
4	1229	27.5	39.5	406	5	Valves placed at each end of potassium filled tube.

Notes:

1. Auxiliary capsule exposed in a vacuum at 1400°F for 100 hr.
2. Cb-1Zr alloy reaction capsule one-in. in diameter x 6 in. long.
3. Initial analyses of zirconium (ppm):

Sample	O	N	H	C
a	638	19	18	---
b	840	14	25	70
<u>c</u>	<u>587</u>	<u>31</u>	<u>40</u>	<u>135</u>
Av.	688	21	28	103
4. Auxiliary capsule No. 3 contained 8.9705 gm potassium, 5.8035 gm zirconium (20 in.² surface area).
5. Analysis of oxygen, nitrogen, and hydrogen in zirconium by vacuum fusion techniques.
6. Potassium from Shipping Container No. 137 slagged, filtered, and hot trapped.

Table III. Potassium Analyses by Zirconium-Gettering Technique
(Potassium sampled during filling of AS-55 alloy capsules Nos.
11 and 12 and D-43 alloy capsules Nos. 2 and 4)

Auxiliary Capsule No.	Zr Analyses,			K Impurities (Calcd.),
	ppm			ppm
	O	N	H	O
7	816	11	11	22

Notes:

1. Auxiliary capsule exposed in a vacuum at 1400°F for 100 hr.
2. Cb-1Zr alloy reaction capsule one in. in diameter x 10 in. long x 0.080-in. thick wall.
3. Initial analyses of zirconium (ppm):

Sample	O	N	H	C
a	638	19	18	---
b	840	14	25	70
c	587	31	40	135
Av.	688	21	28	103
4. Auxiliary capsule No. 7 contained 30.2 gm potassium, 5.1427 gm zirconium (20 in.² surface area).
5. Analysis of oxygen, nitrogen, and hydrogen in zirconium by vacuum fusion techniques.
6. Potassium from Shipping Container No. 137 slagged, filtered, and hot trapped at MSA and re-hot trapped at General Electric for 200 hr at 1300°F.

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